VOL. XVI. 1909

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OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

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VOLUME XVI

SIXTEENTH GENERAL MEETING

NEW YORK CITY, OCTOBER 28, 29, 30, 1909.

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1909

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TABLE OF CONTENTS

PAGE

Group Photograph at Sixteenth General Meeting.....Frontispiece
Proceedings of the Sixteenth General Meeting..... 1
List of Members and Guests in Attendance..... 5
Members Elected and Qualified 8
Constitution of the Society..... 14
By-Laws and Sundry Regulations..... 21
Alphabetical List of Members 28
Geographical Directory of Members..... 53

Recent Advances in Electrochemical Analysis—Dr. Edgar F. Smith.... 65
A New Electrolytic Method for the Preparation of Explosive Antimony—R. C. Palmer and L. S. Palmer..... 79
The Practical Conductance of Electrolytes—Jos. W. Richards and Walter S. Landis 85
Certain Characteristics of Dry Cells—Chas. F. Burgess and Carl Hambuechen 97
Dry Cell Tests—F. H. Loveridge ,.....109
Power for Electrolytic Copper Refining—W. L. Spalding.....125
The Manufacture of Air-Saltpeter by the Process of the Badische Anilin and Soda-Fabrik—Dr. Schönherr 131
Preparation and Properties of Pure Boron—E. Weintraub.....165
The Preparation of Calcium—Francis C. Frary and Walter L. Badger..185
Preparation and Properties of Calcium Carbide—M. DeKay Thompson.197
The Preparation of Silundum—S. A. Tucker, H. F. Kudlich and E. M. Heumann 207
The Titanium Arc—Wm. S. Weedon.....217
Contribution to the Electric Smelting of Iron Ore—E. R. Taylor.....229
Physical Properties of Iron-Copper Alloys—Chas. F. Burgess and James Aston 241
Electro-Cementizing. A Process for Simultaneously Annealing, Cleaning and Zinc-Coating Wire—A. Sang.....257
Laws of Electrode Losses in Electric Furnaces—Carl Hering.....265
A New Method of Measuring Mean Themal and Electrical Conductivities of Furnace Electrodes—Carl Hering.....317
Furnace Electrode Losses—C. A. Hansen.....329
Electrode Losses in Electric Furnaces—E. F. Roeber.....363
Memoir on the Rare Metals—Part III. Vanadium—G. Gin.....393
Index 475
Advertisements .....

VOLUME XVI. 1909.

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES OF THE SIXTEENTH GENERAL MEETING OF THE SOCIETY, HELD IN NEW YORK CITY, N. Y., OCTOBER 28, 29, 30, 1909.

(Number of members registered, 161; guests, 190; total, 351.)

SESSION OF OCTOBER 28th.

The meeting was called to order at 1.30 P. M., in the Assembly Hall of the Chemists' Club, 108 W. 55th Street, President L. H. Baekeland in the Chair.

Papers by W. S. Weedon, of Wilmington, Del.; E. Weintraub, of West Lynn, Mass.; S. A. Tucker, H. F. Kudlich and E. M. Heuman, of Columbia University (read by Prof. Tucker), were read and discussed, as printed in full in these Transactions.

Prof. Edgar F. Smith, of the University of Pennsylvania, followed with an address delivered by special invitation of the Board of Directors, on "Recent Advances in Electrochemical Analysis." The address is printed in this volume. Prof. Henry Armstrong, of England, who was present, closed the session with some appropriate remarks.

During this afternoon the visiting ladies were entertained at tea by Mrs. W. H. Nichols.

FRIDAY, OCTOBER 29th.

The whole of this day was given up to an excursion of inspection of the industries of Bayonne, N. J. The plants of the following companies were visited:

Standard Oil Company (oil refineries).

National Sulphur Company (refining of sulphur).

Pacific Coast Borax Company (manufacture of borax and boric acid).

General Chemical Company (Gröndal briquetting plant, the first American installation of this process of briquetting pyrites cinders).

After lunch, on the shore of the Kill von Kull, where the Mayor of Bayonne, Hon. P. P. Garven, welcomed the Society, the following visits were made:

Babcock & Wilcox Company (water tube boilers and adjuncts).

Electro-Dynamic Company (interpole motors for wide speed variation).

Electric Launch Company (submarine vessels, etc.).

Industrial Oxygen Company (Oxy-acetylene welding and cutting apparatus).

In the evening about 180 members, including ladies and guests, dined together at the Hotel Cumberland.

The visiting ladies spent the afternoon on an automobile excursion.

SATURDAY, OCTOBER 30th

The meeting was called to order at 10 A. M., President L. H. Baekeland in the Chair, in Earl Hall, Columbia University.

Papers by R. C. Palmer, of Columbia, Mo. (read by Jos. W. Richards); Carl Hering, of Philadelphia; C. A. Hansen, of Schenectady; E. F. Roeber, of New York City; Carl Hering; Jos. W. Richards and W. S. Landis (read by J. W. Richards), were read and discussed, as printed in full in these Transactions. Papers by F. C. Mathers and Maximilian Toch were presented in abstract, but have been withheld from publication until a more complete presentation may be obtained.

Lunch was then taken in the University Dining Hall, upon the invitation of the New York Section of the Society; before the afternoon session a group photograph was taken upon the steps

of Earl Hall, as shown in the frontispiece of this volume. (Copies, 9 by 13 inches, heavily mounted, may be obtained directly from the photographer, B. F. McManus, 1296 3d Avenue, New York City.)

Papers by F. H. Loveridge, of Chicago; C. F. Burgess and C. Hambuechen, of the University of Wisconsin (read by Prof. Burgess); C. F. Burgess and James Aston (read by Prof. Burgess); E. R. Taylor, of Penn Yan, N. Y.; Dr. C. Schönherr, of Berlin (read by title); W. L. Spalding, of Buffalo; A. Sang, of Paris, France (read by Jos. W. Richards); M. deK. Thompson, of Massachusetts Institute of Technology (read by Jos. W. Richards); F. C. Frary and W. L. Badger, of the University of Minnesota (read by title), were presented and discussed.

After the discussion upon "Dry Cells," Mr. Hering moved that the Board of Directors be requested to consider the question of appointing a committee to investigate the testing and rating of dry cells and report their recommendations to the Society. The motion was seconded and passed.

Mr. Edward R. Taylor introduced a resolution and a motion which had been originally presented to the Board of Directors at its meeting October 29th, and by it referred to the general meeting with its approval. The resolution and motion were duly seconded and passed. They read as follows:

RESOLUTION.

WHEREAS, We, the members of the American Electrochemical Society are deeply interested in the conservation of water for power purposes and in the mineral resources of our greatly favored land, for the large chemical and electrochemical industries now in existence and which are sure to develop in the near future beyond any possible present estimate, and which are largely dependent upon really cheap power for their highest development, and

WHEREAS, We cannot but view with alarm the possibility of any private interests securing advance control of water rights (coal and other mines of valuable minerals) in the public lands of the United States, and of the several States;

Resolved, That we therefore respectfully urge such legislation as may be deemed wise to meet such peculiar exigencies, as quickly as may be consistent with adequate and proper legislation, in order to protect the public from having to pay extortionate prices or costs for power such as would be inimical to the true prosperity of our industries, and to

encourage new industries in which cheap power is the most important factor.

We desire especially to commend the work of the National Conservation Commission, and to respectfully urge such legislation as may be necessary to make the work of the above commission, and of the Inland Waterways Commission, the most effective for the good of our whole people.

We advise that a copy of this resolution be forwarded to the Hon. William H. Taft, the President of the United States, and that the resolution be printed and copies sent to the members of his Cabinet, to the members of the Senate and House of Representatives, and to the National Conservation and Inland Waterways Commissions.

MOTION.

The American Electrochemical Society, having a deep interest in the conserving of the water resources of the State, commends the work and plans of the New York State Water Supply Commission, and especially urges such legislation as shall enable said commission to carry out the water supply and power developments proposed by it for the Genesee, Hudson and Raquette Rivers, as quickly as practicable, for utility and as object lessons for the whole country and for the world.

As one means to this end, we urge an amendment to the Constitution of the State of New York which will enable said commission to cut timber and make reservoirs in such reserve lands belonging to the State as such commission may recommend. Also that in the interests of proper utility, fallen timber and ripe standing timber may be removed and utilized, according to the most enlightened practice of forestry. We consider that to allow timber to grow to perfection, fall to the ground and decay, is a travesty on correct forestry, and not *conservation* within any proper meaning of the term, holding that true use of "conservation" is to utilize at the right time.

We therefore respectfully urge such legislation and changes in the Constitution of the State of New York as may be necessary in the premises.

We advise that a copy of these recommendations be forwarded to the Governor of the State of New York, and printed copies thereof to the members of the New York State Legislature, to the Governors of the several States, and their respective Water Supply Commissions.

On motion of Mr. Carl Hering, a vote of thanks was directed to be conveyed to the following companies and persons who have aided so materially and effectively in promoting the success of the Sixteenth General Meeting of the Society:

The Standard Oil Company, the National Sulphur Company, the Pacific Coast Borax Company, the General Chemical Com-

pany, the Babcock & Wilcox Company, the Electro-dynamic Company, the Electric Launch Company, the Industrial Oxygen Company, the Chemists' Club, the authorities of Columbia University, the New York Section of the American Electrochemical Society, the Ladies' Committee and the Local Committee—particularly its Chairman and Active Secretary, and the Chairmen and Secretaries of the various sub-committees.

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November 21, 1908.

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J. Rattray Wilson, Niagara Falls, N. Y.
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March F. Chase, Depue, Ill.
Franklin P. Wood, Deadwood, S. D.
Peter Kirkegaard, Deloro, Ontario.
Seabury C. Mastick, New York City.
Algernon K. Johnston, New Brighton, N. Y.
Robert Turnbull, St. Catherines, Ontario.
Richard Merton, New York City.
Fred E. Winslow, Des Moines, Ia.
Edward Randolph, New York City.
G. K. Herzog, Niagara Falls, N. Y.
Robert W. Davis, Jr., Haskell, N. J.
Edward Gudeman, Ph.D., Chicago, Ill.

Albert H. Hooker, Niagara Falls, N. Y.
Frederick Keffer, Greenwood, B. C.
Roy Wells, Divide, Montana.
Edgar D. Stone, Boma, Putnam Co., Tenn.
Drew Haven Dunn, Pasadena, Cal.
Arthur De Wint Foote, North Star Mines, Grass Valley, Cal.
John Elmer King, Corsicana, Tex.
Oscar T. D. Brandt, Tacoma, Wash.
Marcus White, M.E., Silver City, Owyhee Co., Idaho.
Charles Van Brunt, Schenectady, N. Y.
Gustav Aminoff, Baltimore, Md.
Arthur L. Walker, E.M., New York City.
William Wilke, Buffalo, N. Y.
John Johns, Baltimore, Md.
Karl Georg Frank, Ph.D., New York City.
Stephen P. Sharples, S.B., Boston, Mass.
John Thompson, New York City.
Edwin W. Rouse, Jr., Baltimore, Md.
Ralph A. Watson, Mexico City, Mexico.
Raymond Beignet, El Oro, Mexico.
Robert B. Gage, Trenton, N. J.
H. A. Hornor, Riverton, N. J.
Thomas Rowland, Niagara Falls, Canada.
Ellwood S. Harrar, Painesville, O.
K. F. Cooper, Niagara Falls, Canada.
Andrew H. Patterson, A.M., Chapel Hill, N. C.
Joseph F. Body, Philadelphia, Pa.
John W. Dougherty, B.S., Steelton, Pa.
Walter Reichel, Dr. Engr., Berlin, Germany.
H. R. Allensworth, Columbus, O.
John T. Morrow, M.E., New York City.
G. W. Morden, Toronto, Ontario.
Charles Henry Davis, South Yarmouth, Mass.
Chester L. Proebstel, Portland, Ore.
Donald F. Campbell, London, England.
Dorsey A. Lyon, Heroult, Shasta Co., Cal.
Edward P. Jennings, Salt Lake City, Utah.

March 27, 1909.

Charles D. Montague, Wellington, Wash.
W. P. Lass, Treadwell, Alaska.
John H. Pearce, Elkhorn, Mont.
C. Francis Jenkins, Washington, D. C.
John T. Reid, Lovelock, Humboldt Co., Nev.
George R. Rayner, Niagara Falls, N. Y.
Thomas Cantley, New Glascow, Nova Scotia.
Walter M. Saunders, Providence, R. I.
J. V. E. Tate, Kankanhalli, Mysore, India.
Hugo Du Bois, New York City.
C. G. Stoll, New York City.
Juan D. Villarello, Mexico City, Mexico.
George W. Stone, Echota, Niagara Falls, N. Y.
Ernest P. Humbert, Welland, Ontario, Canada.
E. E. White, Fairmount, W. Va.
Walter H. Miller, Orange, N. J.
Albert F. Wurth, Orange, N. J.
Frank Baird, Campo Seco, Calaveras Co., Cal.
Arthur P. Scott, Schenectady, N. Y.
Harry N. Cole, Ann Arbor, Mich.

Hjalmar Ljungh, Baku, Caucasus, Russia.
Anthony Victorin, New York City.
W. G. Abbott, Jr., Lynn, Mass.
Archibald A. C. Dickson, Rejouli P. O., via Nawadah, India.
Milo W. Krejei, Great Falls, Mont.
Samuel E. Hitt, Buffalo, N. Y.
Montgomery Waddell, New York City.
Clarence V. Smythe, Calcutta, India.
James W. Mills, Granite City, Ill.
Edward J. K. Mason, Pittsburgh, Pa.
Leo Daft, Rutherford, N. J.

April 24, 1909.

Chad Herbert Humphries, Watsessing, N. J.
Irenee Du Pont, Wilmington, Del.
Julius E. Ober, Allegheny, Pa.
Napoleon Petinot, Niagara Falls, N. Y.
Clement L. Webster, Charles City, Floyd Co., Iowa.
Kwong Yung Kwang, Lin-tcheng-sien, Pet-chili, North China.
A. Douglas Nash, Flushing, N. Y.
F. Pereira Gamba, Pasto, Republic of Columbia.
Henry T. Murray, Kelvin, Arizona.
Henry A. Gordon, Ben Lomond, New Zealand.
William R. Chedsey, Moscow, Idaho.
H. C. Cridland, Dayton, O.
Howard McClenahan, Princeton, N. J.
F. A. Johnston, Prince Bay, N. Y.
Chas. T. Hennig, Brooklyn, N. Y.
Simon S. Martin, Sparrows Point, Md.
A. J. Woodworth, Sparrows Point, Md.
R. H. Wolff, New York City.
H. B. L. Gorman, Madison, Wis.
Carl D. Hart, New York City.
Fred B. Wheeler, Syracuse, N. Y.
Thomas Boles Allen, Niagara Falls, N. Y.

May 29, 1909.

Edwin H. Smythe, Chicago, Ill.
F. A. Dalburg, Manila, P. I.
F. K. Vial, Chicago, Ill.
Herbert C. Harrison, Lockport, N. Y.
William Ross, Lockport, N. Y.
F. M. De Beers, Chicago, Ill.
Robert Sticht, Queenstown, Tasmania, Australia.
J. F. Fahy, Wellington, N. Z.
Henry K. Richardson, State College, Pa.
John C. Pennie, New York City.
L. E. Howard, Chicago, Ill.
J. A. Seede, Schenectady, N. Y.
G. R. Girwood, Montreal, Canada.
E. G. Love, New York City.
A. L. Simmers, Niagara Falls, N. Y.
C E. Atwood, Iquique, Chile, S. A.
James R. Withrow, Columbus, O.
Harold Jones, Villa Nova de Lima, Minas Geraes, Brazil.
William A. Clarke, Toledo, O.
C. W. Danforth, Sharon, Pa.
S. S. Wales, Munhall, Pa.
A. L. Sonnhalter, Sharon, Pa.
Charles Engelhard, New York City.
Milton W. Franklin, Schenectady, N. Y.
Milton M. Kohn, New York City.
George S. Page, Pittsburgh, Pa.
Philo Kemery, Pittsburgh, Pa.
Radclyffe Furness, Philadelphia, Pa.
C. A. Buck, South Bethlehem, Pa.
Chas. M. Rogers, Newark, N. J.
F. A. Weymouth, Sparrows Point, Md.
Gustavus Hinrichs, St. Louis, Mo.
Alfred H. Barnes, Columbus, O.
A. W. Strong, New York City.

J. Armstrong Rawlins, New York City.
William F. Meredith, Niagara Falls, N. Y.
Wm. R. Palmer, Bridgeport, Conn.
J. R. Cain, Washington, D. C.
C. M. Edward Schroeder, Rutherford, N. J.
L. V. Redman, B.A., Trinity College, Toronto.
A. L. Marsh, Detroit, Mich.
Edward C. Spurge, Niagara Falls, N. Y.
E. P. Coleman, Buffalo, N. Y.
R. H. Stevens, Munhall, Pa.
Henry E. K. Ruppel, South Boston, Mass.
Wm. E. Nickerson, Cambridge, Mass.
Louis Simpson, Fitzroy Harbor, Ont., Canada.
Fernley H. Banbury, Niagara Falls, N. Y.
Joseph F. Becker, Brooklyn, N. Y.
Adolphe Chalas, Paris, France.
P. P. Reese, Munhall, Pa.
Rees James, Munhall, Pa.
C. R. Cary, Philadelphia, Pa.
James Graham, Niagara Falls, N. Y.
John A. Mathews, Ph.D., Sc.D., Syracuse, N. Y.
J. D. Lewis, Braddock, Pa.
E. Friedlaender, Braddock, Pa.
R. C. Reed, Duquesne, Pa.
Chas. E. Taylor, Penn Yan, N. Y.
Brent Wiley, Pittsburgh, Pa.
D. J. Jenkins, Milton, Pa.
William Brady, Chicago, Ill.
H. Hughart Laughlin, Pittsburgh, Pa.
Jesse Coates, B. E., Coatesville, Pa.
G. Ornstein, Niagara Falls, N. Y.
Fred. Crabtree, Pittsburgh, Pa.
H. N. Mueller, Pittsburgh, Pa.
R. S. Orr, Pittsburgh, Pa.
Donald MacKnight Hepburn, Niagara Falls, N. Y.
F. D. Carney, Steelton, Pa.
E. I. Reilly, Jr., Niagara Falls, N. Y.
Harry P. Mills, Toronto, Canada.
Harry L. Brinker, Youngstown, O.
H. M. Lane, Cleveland, O.
S. B. Koch, Palmerton, Pa.
John Hay, New York City.
Benzo Katsura, Tokyo, Japan.

June 25, 1909.

H. V. Vrooman, Mexico City, Mexico.
Saul Dushman, Toronto, Can.
Aaron Merzbacher, Pittsburgh, Pa.
Edwin M. Chance, Pottsville, Pa.
John W. Ostheimer, Paris, France.
Walter H. Scott, Syracuse, N. Y.
Ch. A. Keller, Livet, Isere, France.
Eben B. Clarke, McKeesport, Pa.
Frederick Maeulen, Prince Bay, Richmond Co., N. Y.
E. R. Cole, Niagara Falls, N. Y.
W. A. Ferguson, Mexico City, Mexico.
Clinton W. Lytle, Sharon, Pa.
Belisario Diaz-Ossa, Santiago, Chile, S. A.
H. M. Chance, Philadelphia, Pa.
M. J. Johns, Port Kemble, New South Wales.
L. H. Duschak, Corning, N. Y.
George S. Warren, Sharon, Pa.

July 30, 1909.

Alf. Geo. Jackson, Brisbane, Queensland, Australia.
S. C. Lind, Ann Arbor, Mich.
Gay M. Freeman, Thermopolis, Wyo.
S. Kunitomo, Takata, Echigo, Japan.
Taketaro Takahashi, Yoshida, Japan.

John G. Fairchild, Elizabeth, N. J.
Dr. Willis E. Everette, Tacoma, Wash.
Arthur C. Downes, Fostoria, O.
Yoshikiyo Oshima, Dr. Ing., Tokyo, Japan.

August 27, 1909.

Erwin S. Sperry, Bridgeport, Conn.
G. H. Clevenger, Palo Alto, Cal.
John E. Boesch, E.E., Mexico City, Mexico.
Chas. F. Scott, Pittsburgh, Pa.
L. K. Armstrong, Spokane, Wash.
Ernest C. McKelvy, Washington, D. C.
Raymond L. Lunt, Chicago, Ill.
Georg Hjalmar Johansen, Vindeeren, Christiana, Norway.

September 25, 1909.

Ernesto Stassano, Turin, Italy.
George E. Folk, Chicago, Ill.
Frank Bailey, Perth Amboy, N. J.
Harold J. Wrape, East St. Louis, Ill.
Stanley Cornthwaite, Jossingford, Norway.
Fred S. Pearson, New York City.
Godwin Werliin, Moore, Delaware Co., Pa.
Frank R. McBerty, New York City.
Percy Fawcett, Sheffield, England.
Allen M. Yonge, San Mateo, Costa Rica, Central America.
James Oddie, Ballarat, Victoria, Australia.
Robert H. Buckie, Mechanicville, N. Y.
John B. Huffard, Glen Ferris, W. Va.

October 28, 1909.

Henry A. Gardner, Philadelphia, Pa.
Karl O. E. Olausson, Trollhattan, Sweden.
Stewart J. Lloyd, University, Ala.
Charles Evans, Riverton, N. J.
William L. Robb, Troy, N. Y.
Israel Hatch, Elgin, Ill.
Geo. W. Watts, Toronto, Can.
Wallace P. Cohoe, Toronto, Canada.
Charles W. Gray, Ph.D., Sinnamahoning, Pa.
George E. Marsh, Chicago, Ill.
Raymond W. Baker, New York City.
J. A. Kaemmerer, Toronto, Canada.

CONSTITUTION

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY.

Article I.

Name and Objects.

1. The name of this association shall be the American Electrochemical Society.

2. Its objects shall be the advancement of the theory and practice of Electrochemistry. Among the means to this end shall be the holding of meetings for the reading and discussion of professional and scientific papers on this subject, the publication of such papers, discussions and communications as may seem expedient, and coöperation with chemical, electrical and other scientific and technical societies.

Article II.

Membership.

1. There shall be only one class of members.

2. The only requirement for eligibility to nomination as member is that the candidate shall be interested in the subject of electrochemistry.

Article III.

Admission and Dismissal of Members.

1. Those who have, prior to April 6, 1902, notified the proper authorities that they wish to become members of the Society, are by the adoption of this constitution elected as charter members. They shall not be required to pay an entrance fee; they shall, however, be required to pay their dues for the current year within three months.

2. Application for admission after April 6, 1902, shall be made in writing in a form prescribed by the Board of Directors, and shall refer to at least two members. If the applicant is unable to do this he shall refer to at least three persons, including their address, of reputable standing, preferably electrochemists, chemists, electrical engineers, professors or instructors in institutions of learning. The application shall also state the present occupation of the applicant, and briefly in what capacity he is interested in the subject of electrochemistry.

3. The Secretary shall send to each director, at least twenty days before an election, the names and addresses of the applicants and of their respective references, provided the applications are in due form. The Board of Directors at a duly called meeting shall consider such applications. The election to membership shall be by a vote of the Board of Directors at that or at a subsequent meeting. Proxy votes shall be included; also mail votes received before the election. Two negative votes shall exclude a candidate. The Board of Directors may refuse to elect a candidate whose character, reputation, or professional conduct would make him, in the opinion of the Board of Directors, an undesirable member. The names of those only who have been elected shall be announced to the Society. A candidate who has been duly elected shall be placed on the rolls and have all the rights and privileges of a member as soon as his entrance fee and dues for the current year have been paid.

4. A member desiring to resign shall send a written communication to the Secretary, who shall accept and announce the resignation provided the dues and other indebtedness of that member have been paid to the mailing of his resignation.

5. Upon the written request of ten or more members that, for cause stated therein, a member be dismissed, the Board of Directors shall consider the matter, and if there appears to be sufficient reason, shall advise the accused of the charges against him. He shall then have the right to present a written defense, and to appear in person before a meeting of the Board of Directors, of which meeting he shall receive notice at least twenty days in advance. Not less than two months after such meeting, the Board of Directors shall finally consider the case, and if in the

opinion of the majority of the Board of Directors a satisfactory defense has not been made, and the accused member has not in the meantime tendered his resignation, he shall be dismissed from the Society.

ARTICLE IV.

Dues.

1. The entrance fee, payable on notification of election, shall be five dollars. (Charter members shall be exempt from the payment of the entrance fee.) If not paid within three months after notification, the election of the applicant is thereby canceled.

2. The annual dues shall be five dollars. They shall be due at the beginning of the calendar year, and notice thereof shall be mailed to each member before the end of the preceding year. If not paid before the sixteenth day of March, the delinquent member shall lose his right to vote or to receive the publications of the Society, until said dues are paid. Members will pay dues for, and receive the publications of, the year in which they are elected, but if elected within two months of the end of the calendar year may, at their option, pay for, and enter with, the next coming year.

3. Any member who, at the sixteenth day of March, is in arrears for two years shall be dropped from the rolls, and shall not be included in subsequently published lists of members. The Board of Directors may reinstate a member who has been dropped, provided all his arrears have been paid.

ARTICLE V.

Officers.

1. The officers of the Society shall be a President, six Vice-Presidents, nine Managers, a Secretary, and a Treasurer, who shall hold office as follows:

2. The President, the Secretary, and the Treasurer for one year, the Vice-Presidents for two years, and the Managers for three years. At each annual meeting a President, three Vice-Presidents, three Managers, a Secretary, and a Treasurer shall be elected, and their term of office shall begin at the close of the

annual meeting, except during the year 1902, when it shall begin with their election. Each officer shall hold office until his successor has qualified.

3. A vacancy in the office of President shall be filled by the senior Vice-President; a vacancy in the office of Vice-President shall be filled by the senior Manager. Seniority between officers of the same rank and date of election shall be determined by the number of votes received when they were elected; or if the same, then by the date of their election to membership, or if this be also the same, then in alphabetical order. All other vacancies shall be filled by the Board of Directors.

4. No officer shall receive directly or indirectly, any salary, compensation or emolument from the Society, either as such officer or in any other capacity, or be interested, directly or indirectly, in any contract relative to the operations conducted by the Society or in any contract for furnishing supplies thereto, unless authorized either by the by-laws or by a vote of the majority of the entire Board of Directors.

Article VI.

Election of Officers.

1. The Secretary shall send to all members nomination blanks not later than the first of January, and nominations, not necessarily upon these forms, will be received by the Secretary not later than the first of February. He shall mail to every member before February 16th a printed list of all candidates thus nominated who have received more than 5 per cent. of the number of valid nominating ballots cast. The voting need not be restricted to the names on this list. The Board of Directors may endorse those which it prefers, and if so, the published list shall state it. The voting shall be by secret letter ballot, the voter shall sign his name on an outside envelope, and the ballot shall be inclosed in an inner sealed and unmarked envelope. Ballots must reach the Secretary before the sixteenth day of March. The results of the election shall be announced at the annual meeting.

2. At the Directors' meeting, to be held in February or March, the Board of Directors shall appoint three tellers, who shall not

be members of the Board of Directors, and three alternates. Should this Directors' meeting not be held, the President shall appoint these tellers, who shall be notified by the Secretary.

3. The tellers shall meet as soon after the fifteenth day of March as possible. The Secretary shall deliver to the tellers all ballots received by him, marking the outside envelopes received from members not entitled to vote. The tellers shall open only the envelopes not thus marked, and shall proceed in secret to count the votes received before the sixteenth day of March. All ballots and unopened envelopes shall be returned to the Secretary, who shall preserve them for one month. At the end of one month the Secretary shall return to the senders all rejected envelopes and shall open all unidentified envelopes. Cumulative votes shall count as one vote. The tellers shall prepare and sign a report of the results of the vote, which shall be sealed, and shall remain in the possession of the Chairman of the Committee, who shall hand this report to the presiding officer at the ensuing annual meeting. The presiding officer shall then cause this report to be read, and shall declare duly elected the eligible persons receiving the greatest number of votes for the respective offices.

4. The President, Vice-Presidents and Managers shall, after the expiration of the full term of office to which they were elected, be ineligible for immediate re-election to the same office.

Article VII.

Management.

1. The affairs of the Society shall be managed by a Board of Directors under this constitution and by-laws. This board shall consist of the officers and the two junior past presidents. The President, Vice-President, Secretary, and Treasurer of the Society shall hold the same offices in this Board of Directors.

2. The Board of Directors shall prepare and adopt a series of by-laws which shall govern its meetings and its procedure, as also that of the Society under this constitution. It shall require a two-thirds vote of the whole board to adopt or amend these by-laws; proxy and mail votes shall be counted when received before the

vote is counted. The text of the proposed by-laws or amendments shall be furnished to each member of the Board of Directors at least ten days before the meeting at which the vote on the same is to be taken.

3. The Board of Directors may delegate any or all of its powers, except in those cases in which this constitution requires a vote of the whole board, to an Executive Committee of not less than one-third of the number of members of the Board of Directors. The President and Secretary shall be members of this Executive Committee. The committee shall conduct the affairs of the Board of Directors between its meetings. All members of the Board of Directors shall receive notice of the meetings of this Executive Committee and shall have the right to attend and vote. A majority of the Executive Committee shall constitute a quorum.

4. At the request of the President, matters requiring action by the Board of Directors may be discussed and voted upon by a mail vote of the Board of Directors.

5. The duties of the officers shall be such as usually pertain to the offices they hold, besides any others designated in the constitution or by-laws or by the Board of Directors. The Secretary shall, under the direction of the President and Board of Directors, be the executive officer of the Society. The Secretary and the Treasurer shall present reports annually to the Board of Directors, before the annual meeting. The Board of Directors shall present a report annually to the Society at its annual meeting. The nature of these reports shall be specified in the by-laws.

6. All standing committees shall be directly responsible to the Board of Directors, and shall be appointed by the President or by the Board of Directors.

Article VIII.

Meetings.

1. The annual meeting shall be held during the last week of March or in April. Business affecting the organization of the Society shall be transacted only at the annual meetings. Proxy or mail votes shall be received.

2. Other meetings may be held at such times and places as the

Board of Directors shall select. The President shall call a meeting at the written request of 30 or more members.

3. Notice of all ordinary meetings shall be sent to all members at least twenty days in advance, and of the annual meeting, thirty days in advance.

Article IX.

General.

1. A quorum of the Society shall consist of one-tenth the number of members residing in the United States, present in person or by proxy.

2. Every member entitled to vote at any meeting may vote by proxy or, when the subject-matter has not been changed, by mail. No proxy shall be valid after the expiration of eleven months from the date of its execution. Every proxy shall be revocable at the pleasure of the person executing it.

3. The fiscal year of the Society shall begin January 1st.

Article X.

Amendments.

Proposals to amend this constitution shall be made in writing to the Board of Directors and signed by at least ten members; they shall reach the Secretary not later than February 1st. The Board of Directors shall consider them and may amend them. The original, with the action of the Board of Directors, shall then be presented to the Society at its annual meeting, for final action. A majority vote shall decide, the proxies being included, provided that a quorum is represented, in person or by proxy.

BY-LAWS.

1. *Order of Business.*—At the annual meeting of the Society the order of procedure of the business session shall be as follows:

Reading of the minutes of last meeting.

Report of the Board of Directors, including those of the Secretary and the Treasurer.

Report of Standing Committees.

Deferred business.

New business.

Report of tellers of election.

Address of the retiring president. (This may be deferred to a later session of the general meeting, at the discretion of the framers of the program of the meeting.)

The business meeting of the Society shall preferably be the second day of the annual meeting.

2. *Meetings of the Board of Directors.*—The Board of Directors shall hold a regular meeting immediately preceding each general meeting of the Society, or at the annual meeting at any time before the business meeting of the Society.

Between said meetings the Board of Directors may hold special meetings when called by the President, and coincident with monthly meetings of the Executive Committee when applications for membership are to be acted upon.

At the regular meetings of the Board of Directors the order of procedure shall be as follows:

Reading of the minutes of the last regular meeting and of any special meeting, the minutes of which have not been approved.

Election of new members.

Appointment of tellers of election.

Approval of bills.

Communications from the Secretary.

Communications from the Treasurer.

Communications from the President.

Report of committees.
Deferred business.
New business.
Adjournment.

At special meetings of the Board of Directors, the order of procedure shall include the reading of the minutes of the last meeting of the Board and such other of the above items of procedure in their order given as are to be acted upon.

3. *Duties of Officers.*—The President shall preside at all meetings of the Society and at those of the Board of Directors. He may represent the Society at any function to which no other particular delegate has been appointed by the Society or the Board of Directors. The President (or any director) shall have the power of appealing any action of the Executive Committee to the Board of Directors, within two weeks of its adoption, which action can then be carried into effect only after approval by a majority of the entire Board of Directors voting on said explicit action either in person or by mail ballot. He shall deliver an address at the annual meeting of the Society at which his term of office expires.

The Vice-Presidents, in the order of their seniority, shall assume the duties and responsibilities of the President in case of his death, disability or absence from fulfilling his duties.

The Secretary, besides performing the usual duties of that office, shall be the guardian of the records of the Society, collect dues from members and all bills owing to the Society, and pay such amounts to the Treasurer. At meetings of the Society he shall collect and receive manuscripts of the papers read, with drawings or diagrams thereto belonging, and shall be responsible for their custody until he hands them to the Publication Committee. He shall report to the Board of Directors at its meeting held at the annual meeting of the Society, on the condition of the affairs of the Society, such as the membership, publications, number of meetings, number of papers read and in general a summary of the work and condition of the Society, excepting its finances, and make such recommendations as he thinks will facilitate the transaction of the business affairs of the Society or in any way advance its interests.

The Treasurer shall report to the Board of Directors at its

meeting immediately preceding the business session of the annual meeting of the Society on the condition of the finances of the Society and make such recommendations as will in his opinion tend to safeguard or improve the same.

The Board of Directors shall report to the annual meeting on the general affairs of the Society, including the abstracts of the reports of the Secretary and the Treasurer and any other matters which they may consider of interest to the Society.

4. STANDING COMMITTEES.—*Committee on Papers.*—A Committee on Papers, consisting of at least five members, shall be appointed annually by the incoming president and approved by the Board of Directors, and shall procure and pass upon all papers for the meetings for one year, beginning with the meeting following the annual meeting.

Publication Committee.—A Publication Committee, consisting of at least five members, shall be appointed annually by the incoming President and approved by the Board of Directors, and shall have charge of all publications for one year beginning with the transactions of the meeting following the annual meeting.

The Publication Committee shall receive from the Secretary the manuscripts of papers read before the Society, with illustrations or diagrams pertaining thereto and the discussion thereon, and shall publish the transactions of the Society. It shall have power to decide on the fitness of such matters for publication and in consultation with the authors to edit such matter, so that its publication may serve the best interests of the Society. It may edit the discussion so as to give it proper literary and parliamentary form, but not, however, changing its substance and meaning without the consent of the author.

The President and Secretary shall be members *ex-officio* of all standing committees.

5. *Quorum of Directors.*—A quorum of the Board of Directors shall be twelve, present in person or by proxy.

6. *Treasurer's Accounts.*—The President shall appoint two auditors, at least one week before the annual meeting, to audit the accounts of the Treasurer. At least one of the said auditors shall be a member of the Board of Directors, and said member shall

report the result of said audit to the Board of Directors at its meeting, preceding the annual business meeting of the Society.

7. *Entrance Fees.*—Any entrance fee paid by an applicant shall be returned if the applicant is not elected.

8. *Guests at Meetings.*—Guests may be introduced to the meetings of the Society by members and may be given the privilege of attending the meetings and may be given such further privileges as may be extended to them by the Local Committee or the President or Secretary.

9. *Membership Committee.*—A Committee on Membership, whose function shall be to enlarge by systematic effort the membership of the Society, shall be appointed annually by the incoming President. It shall consist of 15 or more members.

Regulations Concerning the Formation and Conduct of Local Sections.

1. A Local Section of this Society may be authorized by the Board of Directors, at the written request of three members. Only one Section shall be authorized in any one locality.

2. Any member of the Society, in good standing, may enroll as member of any Local Section, provided he complies with the regulations of the said Section. Only members of this Society are eligible to membership in its Local Sections.

3. The regulations of Local Sections are subject to the approval of the Board of Directors.

4. The expenses of each Local Section shall be borne by the members enrolled in said Section. The Society shall not be responsible for any debts contracted by a Local Section or the officers thereof.

5. The Society will print advance copies of papers offered to Local Sections, in order to facilitate full discussion thereon, provided that said papers are approved for such advance publication by the chairman of the Local Section and by the Publication Committee of the Society.

6. Papers read before Local Sections, and discussion thereon

if reported, are to be considered as the property of this Society. They shall be submitted to the Publication Committee of the Society and published in the Transactions if approved by it. Reading of a paper before a Local Section does not carry with it the right of publication in the Transactions.

Papers read before Local Sections may be offered for reading at general meetings of the Society, and will be given equal standing with other papers on the programme of said meeting, when approved by the Committee on Papers.

7. The Board of Directors reserves the right to abrogate, amend or add to these regulations, or to pass upon such questions concerning the welfare of Local Sections not covered by these regulations.

8. Each Local Section shall transmit promptly to the Secretary of the Society, an abstract of its proceedings, including the titles and names of authors of all papers read before it, for the purpose of preparing a report to be published in the Transactions of the Society, and for the purpose of enabling the Board of Directors to comply with Articles 3 and 7 of these regulations.

Action of May 2, 1907.

Local Sections of the Society are allowed to draw upon the Society for financial assistance during the current year, to the limiting sum not exceeding $1.00 for each of their members. This money is intended to pay rent for rooms, sending of notices, etc.

[The above action, taken in 1907 for that current year, has been renewed in subsequent years when the Society was able to give Local Sections this assistance.]

Complete Sets of Transactions.

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AMERICAN ELECTROCHEMICAL SOCIETY

DIRECTORY OF MEMBERS

January 1, 1910.

(Members of date April 3, 1902, are charter members.)

ABADIE, Eugene H. (Sept. 26, '08) Consult. Eng., 1720 Third Nat. Bank Bldg., St. Louis, Mo.; res., 5148 Westminster Place.
ABBOTT, William G., Jr. (Mar. 27, '09) Research Engineer, General Electric Co., Lynn, Mass.; res., 54 Park St.
ACHESON, E. G. (Apr. 3, '02) Inter. Acheson Graphite Co., Niagara Falls, N. Y.
ACHESON, Edward G., Jr. (July 31, '08) Pres. Acheson Oildag Co., Niagara Falls, N. Y.
ACKER, Chas. E. (Apr. 3, '02) 1 W. 121st St., New York City.
ACKER, O. E. (Apr. 4, '03) Bourbon, Ind.
ADAMS, Prof. C. A. (Apr. 3, '02) Asst. Prof. of Elec. Eng., Harvard University, Cambridge, Mass.
ADAMS, Dr. Isaac (Apr. 3, '02) 1776 Massachusetts Ave., Cambridge, Mass.
ADAMSON, G. P. (Dec. 4, '02) Gen. Mgr. Baker & Adamson Chem. Co.; res., 233 Reeder St., Easton, Pa.
ADDICKS, Lawrence (Apr. 3, '02) Supt. U. S. Metals Refining Co., Chrome, N. J.; res., 420 N. Broad St., Elizabeth, N. J.
ADSIT, Charles G. (Sept. 26, '08) Consult. Eng., Warren, Ariz.
AHLBRANDT, G. F. (Jan. 29, '09) Open Hearth Supt., Amer. Rolling Mills Co., Middletown, Ohio.
AIKEN, Robert H. (Oct. 10, '03) Consulting Metallurgist, Winthrop Harbor, Ill.
AKERBERG, Olof T. (Oct. 26, '08) Eng. with Langeds Aktienbolag, Langed, Dalsland, Sweden.
ALBRIGHT, Langdon (Sept. 26, '08) Sales Eng., Niagara, Lockport and Ontario Power Co., 816 Fidelity Bldg., Buffalo, N. Y.
ALDEN, John (Apr. 3, '02) Pacific Mills, Lawrence, Mass.
ALDRIDGE, Walter H. (Jan. 28, '08) Consult. Eng., Canadian Pacific R. R., Trail, B. C., Canada.
ALLEN, Thos. B. (Apr. 24, '09) Chemist, The Carborundum Co., Niagara Falls, N. Y.
ALLEN, Wyatt H. (Sept. 4, '02) Consulting Engineer, 910 Union Trust Bldg., San Francisco, Cal.
ALLENSWORTH, H. R. (Feb. 27, '09) Supt. Fire and Police Telegraph, 89 N. Front St., Columbus, Ohio.
ALLYN, R. S. (Feb. 5, '03) Patent Lawyer, 16 Exchange Place, New York; res., 24 Irving Place, Brooklyn, N. Y.
AMINOFF, Gustav (Feb. 27, '09) Chemist, Baltimore Copper Smelting & Rolling Co., Baltimore, Md.
AMSTER, N. L. (Apr. 3, '02) Consult. Min. Eng., 32 Equitable Bldg., Boston, Mass.
APPELBERG, Axel O., Ph.D. (Nov. 26, '07) Kungsportsavengen 22, Gothenburg, Sweden.
ARISON, William H. (July 31, '08) Mgr., Int. Acheson Graphite Co., Niagara Falls, Ont., Canada.
ARMOR, James C. (Mar. 4, '05), Research Div., Eng. Dept., Westinghouse El. & Mfg. Co.; res., 11 Wheeler Ave., Ingram, Pa.
ARMSTRONG, Lyndon K (Aug. 27, '09) Mining Engineer, Editor Northwest Mining News, P. O. Drawer 14, Spokane, Wash.
ARNOLD, T. Herbert (Mar. 4, '05) Miami, Ariz.
ARTH, G. (Jan. 8, '04) Institut Chimique, rue Grandville 1, a Nancy M et M, France.
ASHCROFT, E. A. (May 1, '06) Electrochem. Eng., Vadheim, Sogne, Norway; res., 82 Victoria St., London, S. W., Eng.
ASKEW, C. B. (Apr. 3, '02) Consult. Eng., 948 Monadnock Bldg., Chicago, Ill.
ASTON, James (Jan. 28, '08) Electrochem. Lab., Univ. of Wisconsin, Madison, Wis.; res., Harrison and Ray Sts.
ATWOOD, Carlton E. (May 29, '09) Sales Engineer, Westinghouse Products, Casilla 704, Iquique, Chile, South America, care of T. K. Robinson.
ATWOOD, George F. (Apr. 3, '02) 111 N. 11th St., Newark, N. J.
AUBRY, Henri (Oct. 29, '08) with H. Aubry & Cie, Albertville, Savoie, France.

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AYLSWORTH, J. W. (Mar. 5, '03) Chem. Eng. and Experimenter, 223 Midland Ave., E. Orange, N. J.
BABCOCK, Clifford D. (Oct. 29, '08) Sci. Mgr., United Wireless Telegraph Co., Jersey City, N. Y.; mailing address, 73 Christopher St., New York City.
BABROWSKY, G. (Mar. 25, '08) Sokolsko Str., 16, Prague, Bohemia.
BACHERT, A. (Nov. 27, '09) Chief Engineer, also private engineering, East Broad Top R. R. Iron & Coal Co., Tyrone, Pa.; mailing address, 1458 Logan Ave.
BACON, Ellis W. (Apr. 7, '06) in Pub. Dept., J. B. Lippincott Co., 227 S. Sixth St.; res., 3603 Baring St., Philadelphia, Pa.
BAEKELAND, Dr. Leo (June 6, '03) "Snug Rock," Harmony Park, Yonkers-on-Hudson, N. Y.
BAGOT, C. G. S. (Oct. 29, '08) El. and Min. Eng., Heriot Bay, British Columbia.
BAILEY, Frank (Sept. 25, '09) Factory Manager, Vulcan Detinning Co., Sewaren, N. J.; res., 218 Brighton Ave., Perth Amboy, N. J.
BAIRD, Frank (Mar. 27, '09) Metallurgist, care of Penn. Chemical Wks., Campo Seco., Calaveras Co., Cal.
BAKER, Chas. E. (Aug. 5, '05) Metallurgist, 201 Traders Bldg., Chicago, Ill.
BAKER, Herbert A. (Dec. 27, '07) Chemist, American Can Co., Boston and Hudson Sts., Baltimore, Md.
BAKER, Raymond W. (Oct. 28, '09) Mgr., Research Laboratory, Parker-Clark Electric Co., 135 Broadway, New York City; res., 570 W. 183d St.
BALLWEG, John H. (Sept. 26, '08) General Contractor, Cascade Locks, Ore.
BANBURY, Ferneley H. (May 29, '09) Supt., Acheson Oildag Co., Niagara Falls, N. Y.
BANCROFT, Wilder D., Ph.D. (Apr. 3, '02) Cornell University; res., 7 East Ave., Ithaca, N. Y.
BARKER, E. R. (Apr. 3, '02) 22 Russell St., Arlington, Mass.
BARNES, Alfred H. (May 29, '09) Assistant in Physics, Ohio State University, Columbus, Ohio; res., 325 West 10th Ave.
BARR, B. M. (April 3, '02) 32 W. 40th St., New York.
BARRETT, Jas. M. (Apr. 2, '04) Phipps Power Bldg., Pittsburgh, Pa.
BARSTOW, W. S. (Apr. 3, '02) 56-58 Pine St., New York.
BARTLETT, Frank S. (Jan. 29, '09) Supply Dept., General Electric Co., Schenectady, N. Y.; res., 186 Furman St.
BARTON, C. B. (Sept. 4, '02) Supt. of Electrolytic Bleach Plant, Burgess Sulphite Fibre Co., Berlin, N. H.
BARTON, P. P. (Feb. 2, '06) Gen. Mgr. Niagara Falls Power Co., 352 Buffalo Ave., Niagara Falls, N. Y.
BASKERVILLE, Chas., Ph.D. (Apr. 4, '03) Prof. of Chem., College City of New York, New York City.
BASSETT, Wm. H. (Nov. 26, '07) Chemist, The American Brass Co., P. O. Box 1047, Waterbury; res., Torrington, Conn.
BATES, Wm. S. (Apr. 3, '02) 536 Fulton St., San Francisco, Cal.
BAUER, G. W. (Sept. 4, '93) Vice-Pres. and Chem., Bauer, Schweitzer Hop and Malt Co., 1722 Buchanan St., San Francisco, Cal.
BEAZLEY, Arthur T. (Sept. 26, '08) El. Eng., The California Oilfields, Ltd., Oilfields, Cal.
BECKER, Joseph F. (May 29, '09) Generan Agent, Edison Elec. Illuminating Co. of Brooklyn, Brooklyn, N. Y., 360 Pearl St.
BECKET, Frederick M. (Apr. 3, '02) The El.-Met. Co. of America, Niagara Falls, N. Y.; P. O. Box 158.
BECKMAN, John W. (May 6, '05) American Cyanamid Co., Niagara Falls, Canada.
BEEBE, M. C. (Feb. 6, '04) Univ. of Wisconsin, Madison, Wis.
BEERS DE, F. M. (May 29, '09) Pres. & General Mgr., Swenson Evaporator Co., 945 Monadnock Bldg., Chicago, Ill.
BEIGNET, Raymond (Feb. 27, '09) Chemist, Compania Minera dos Estrellas, El Oro., Mexico; Apartado No. 34.
BENECKE, Adelbert, O. (Sept. 26, '08) Chief Eng., Amer. Inst. Co.; res., 19 Vermont Ave., Newark, N. J.
BENNETT, Edw. (Feb. 6, '04) Electrician, Telluride Power Co., Provo City, Utah.
BENNIE, P. McN. (July 1, '04) FitzGerald & Bennie Laboratories, Niagara Falls, N. Y.
BENOLIEL, Sol. D., B.S., E.E., A.M. (Sept. 4, '02) International Chem. Co., Camden, N. J.
BERG, E. J. (Oct. 2, '02) Univ. of Illinois, Urbana, Ill.
BERRY, Edw. R. (Dec. 1, '06) Chief Chem., Eng. Lab., Gen. Elec. Co., Lynn, Mass.; res., 107 Cross St., Malden, Mass.
BETTS, Anson G. (Apr. 3, '02) head 16th St., N., Troy, N. Y.
BIDDER, VON, Frederick, Dipl. Eng. (Oct. 29, '08) Usines Electriques de la Lonza, Vernier, near Geneva, Switzerland.
BIERBAUM, Christopher H. (Apr. 3, '02) Consult. Eng., Vice-Pres. Lumen Bearing Co., Pres. Robson Smelting Co., 418 Prudential Bldg., Buffalo, N. Y.
BIGELOW, S. Lawrence, Ph.D. (May 9, '03) Jr. Prof. of Gen. and Phys. Chem., Univ. of Mich.; res., 1520 Hill St., Ann Arbor, Mich.
BIJUR, Jos. (Oct. 10, '03) Westinghouse Storage Battery Co., Boonton, N. J.

BIXBY, Geo. L. (Aug. 7, '02) Pope Motor Car Co.; res., 15 East 36th St., Indianapolis, Ind.
BLAKE, Lucien I., Ph.D. (Mar. 5, '04) Submarine Signal Co., 1936 Curtis St., Denver, Colo.
BLEECKER, Warren F. (Nov. 27, '09) Chief Metallurgist, Colorado Vanadium Co., Boulder, Colo.; mailing address, 2235 15th St.
BLOCK, David J. (Sept. 26, '08) Supt. American Metallizing Co., First Natl. Bank Bldg., Chicago, Ill.
BLOCK, W. S. (Oct. 2, '02) Pres. Roberts Chem. Co., 60 Wall St., New York.
BODY, Joseph F. (Feb. 27, '09) Consulting Engineer; Rochambeau Apt., Baltimore, Md., P. O. Box 842.
BOECK, Percy A. (Feb. 28, '08) Chemist, Norton Emery Co., Worcester, Mass.
BOERICKE, Gideon (Mar. 5, '04) Sec. and Treas. Primos Chem. Co., Primos, Del. Co., Pa.
BOESCH, John E., E.E. (Aug. 27, '09) Chief Inspector, Mexican Light & Power Co., Mexico City, Mexico, D. F.
BOGUE, Chas. J. (Apr. 3, '02) Mfr. of Elec. Machinery, 213-215 Center St., New York.
BOIS DU, Hugo (Mar. 27, '09) Secretary, The Roessler-Hasslacher Co., 100 William St., New York City.
BONNA, Dr. Aug. E (Apr. 2, '04) Rue Petitot 15, Geneve, Switzerland.
BOON, Prof. John D. (Apr. 3, '02) Polytechnic College, Fort Worth, Tex.
BOWER, B. Frank (Jan. 29, '09) 2d Vice-President, Henry Bower Chem. Mfg. Co., 29th St., and Grays Ferry Road, Philadelphia.
BOWMAN, Walker (Apr. 3, '02) 39 Cortlandt St., New York.
BRADLEY, C. S. (Nov. 6, '02) Pres. Atmospheric Products Co., 44 Broad St., New York.
BRADLEY, John Clement (Nov. 26, '07) Ass't Chemist, The Amer. Brass Co.; res., 74 Litchfield St., Torrington, Conn.
BRADLEY, Walter E. F. (June 29, '07) 41 Park Row, New York City.
BRADLEY, Walter M. (Apr. 3, '02) Sheffield Scientific School, Min. Lab.; res., 520 Whitney Ave., New Haven, Conn.
BRADY, Wm. Burke (Oct. 28, '09) Assistant Superintendent, National Carbon Co., Cleveland, Ohio; mailing address, 12582 Clifton Boulevard.
BRADY, William (May 29, '09) Chief Chemist, Illinois Steel Co., Chicago, Ill; mailing address, 7642 Marquette Ave.
BRAGDON, W. B. (Nov. 27, '09) Genl. Mgr., Cresumpscot Electric Co., S. D. Warren Co., Westbrook, Cumberland Mills, Me.; mailing address, 64 Lamb St.
BRANDT, Oscar T. D. (Feb. 27, '09) Mgr. Northwestern L. D. Tel. Co., Tacoma, Wash.; Box 24.
BRAY, William C. (May 30, '08) Inst., Mass. Inst. Tech., Room 16 Eng. C., Boston, Mass.
BRECKENRIDGE, James M. (Apr. 30, '08) Student, Univ. of Wis.; res., 608 W. Dayton St., Madison, Wis.
BREED, George (June 3, '04) Consult. Eng., Witherspoon Bldg., Philadelphia, Pa.
BRENEMAN, A. A. (May 1, '06) Analyt. & Consult. Chem., 97 Water St., New York.
BRINDLEY, G. F. (Apr. 3, '02) care of The Mexican Steel & Chem. Co., Apartado 1215, Mexico, D. F.
BRINKER, Harry L. (May 29, '09) Chief Chemist, Carnegie Steel Co., Youngstown, Ohio; res., 322 North Ave.
BRISTOL, Wm. H. (Dec. 1, '06) The Bristol Co., Waterbury, Conn.
BROOKS, Morgan, Ph.D. (Apr. 3, '02) Univ. of Illinois, Urbana, Ill.
BROWN, Harold P. (Apr. 3, '02) Elec. Eng., 120-122 Liberty St., New York, N. Y.
BROWN, J. Stanford (Apr. 3, '02) Vice-Pres. and Treas. New York Realty Owners' Co., 489 Fifth Ave, New York; res., 10 Belmont Ave., Yonkers, N. Y.
BROWN, Dr. John W. (July 31, '07) Director Research and Battery Lab., National Carbon Co., Cleveland, Ohio.; mailing address, 1377 W. 116 St.
BROWN, O. W. (Apr. 3, '02) Associate Prof. in Chem., Indiana Univ., 429 E. Seventh St., Bloomington, Ind.
BROWN, W. G., B.S., Ph.D. (Apr. 3, '02) Prof. of Chem., Univ. of Missouri, Columbia, Mo.
BROWNE, David H. (Apr. 3, '02) Metallurgist, Canadian Copper Co., Copper Cliff, Ont., Can.
BROWNE, Wm. Hand, Jr. (Apr. 3, '02) North Carolina College of Agriculture and Mechanic Arts, West Releigh, N. C.
BRUNT VAN, Chas. (Feb. 27, '09) Chemist, General Electric Co., Research Laboratory, Schenectady, N. Y.
BRYDEN, Chas. L. (Nov. 27, '09) Head of School of Metal Mining & Metallurgy, Int. Corresp. School, Text-book Dept., Scranton, Pa.; res., 436 Colfax Ave.
BUCH, N. W. (Nov. 6, '03) Safety Amorite Conduit Co.; mailing address, West Pittsburgh, Lawrence Co., Pa., Box 97.
BUCHANAN, Leonard B. (Apr. 3, '02) Stone & Webster, 84 State St., Boston, Mass.
BUCK, C. A. (May 29, '09) General Supt., Bethlehem Steel Co., South Bethlehem, Pa.
BUCK, H. W. (May 7, '04) 49 Wall St., New York City.

BUCK, Leon H. (Feb. 2, '06) Nat. Analine Chem. Co., 110 W. 64th St., New York City.
BUCKIE, Robert H. (Sept. 25, '09) Supt., Electrolytic Bleach and Soda Plant; The W. Va. Pulp and Paper Co., Mechanicville, N. Y.
BUCKLEY, Hubert (June 3, '05) R. F. D. Hatboro, Pa.
BURGESS, Prof. C. F. (Apr. 3, '02) Engineering Bldg., Univ. of Wisconsin, Madison, Wis.
BURLING, B. B. (Jan. 5, '07) Electrochemist, Waukegan, Ill.
BURNS, Willis T. (Nov. 6, '03) in charge of Electrolytic Refinery, Boston and Mont. Cons. Cop. S. and Min. Co., Great Falls, Mont.
BURSON, W. W. (Nov. 27, '09) Vice-president, Burson Knitting Co., Rockford, Ill.; res., 6905 Sheridan Road, Chicago, Ill.
BURWELL, Arthur W., Ph.D. (Nov. 5, '04) P. O. Box 1193, Helena, Montana.
BUTTERS, Chas. (July 1, '05) 333 Kearny St., San Francisco, Cal.
BYRNES, Eugene A., Ph.D. (Apr. 3, '02) Patent Lawyer, 918 F St., N. W.; res., 2539 13th St., Washington, D. C.
CAIN, J. R. (May 29, '09) Chemist, The Bureau of Standards, Washington. D. C.
CALBERLA, Roland (Nov. 26, '07) Niederschoenweide bei Berlin, Germany; care of Kunheim & Co.
CALDWELL, Edw. (Apr. 3, '02) 239 W. 39th St., New York; res., 50 Westervelt Ave., Plainfield, N. J.
CALVO, Pedro P. (Nov. 21, '08) Civil Engineer, P. O. Box 26, Bogota, Columbia, South America.
CAMERON, Frank K., Ph.D. (Oct. 7, '05) Chem. in charge of Phys. and Chem. Investigation, Bureau of Soils, U. S. Dept. of Agr., Washington, D. C.
CAMERON, Walter S. (Apr. 3, '02) 239 W. 136th St., New York.
CAMPBELL, Donald F. (Feb. 27, '09) Electrometallurgist, The Royal Societies Club, London, S. W., England.
CANTLEY, Thomas (Mar. 27, '09) Genl. Manager, Nova Scotia Steel & Coal Co., Ltd., New Glascow, Nova Scotia, Canada.
CAPP, J. A. (Sept. 4, '02) Chief of Testing Laboratory, Gen. Elec. Co., Schenectady, N. Y.
CARHART, Henry S., LL.D. (Apr. 3, '02) Univ. of Mich., Ann Arbor, Mich.
CARLSON, BIRGER (Nov. 5, '04) Chemist, Electrochem. Works of Stockholm, Superfosfat Aktiebolag of Stockholm, Sweden, Mansbo, Avesta, Sweden.
CARNEY, F. D. (May 29, '09) Assistant General Superintendent, Penna. Steel Co., Steelton, Pa.
CARRIER, C. F. (Mar. 5, '03) Sewaren, N. J.
CARSE, David B. (Mar. 4, '05) President, David B. Carse & Co., 165 Broadway, New York City.
CARVETH, H. R., Ph.D. (Apr. 3, '02) Works Mgr., Niagara Electrochemical Co., Niagara Falls, N. Y.; res., 118 Buffalo Ave.
CARY, Charles R. (May 29, '09) Engineering Salesman, The Leeds & Northrup Co., 4901 Stenton Ave., Philadelphia.
CARY, Edward E. (Sept. 26, '08) Pres. Edward E. Cary Co., 59-61 Park Place, New York City.
CASE, Willard E. (Oct. 2, '02) Metropolitan Club, 5th Ave. and 60th St., New York City.
CASSELBERRY, Harry (June 29, '07) 2214 7th Ave., Altoona, Pa.
CATANI, Remo (Aug. 31, '07) Electrical Engineer, Societe "Elba," Porto Ferraio, 45 Palazzo Doria, Rome, Italy.
CATLIN, Chas. A. (Nov. 6, '03) Chemist, Rumford Chem. Works, 133 Hope St., Providence, R. I.
CHALAS, Adolphe (May 29, '09) Representative, Keller-Leleux Steel Co., Paris, France; mailing address, 45 Rue Emile Menier.
CHANCE, Edwin M. (June 25, '09) Assistant Chemist, P. & R. Coal & Iron Co., Pottsville, Pa.; res., 313 South 2d St.
CHANCE, H. M. (June 25, '09) Consulting Mining Engineer, Drexel Bldg., Philadelphia.
CHANDLER, Dr. C. F. (Jan. 8, '03) Prof. of Chem., Columbia Univ., New York.
CHASE, March F. (Feb. 27, '09) Genl. Superintendent, Mineral Point Zinc Co., Depue, Ill.
CHEDSEY, William R. (Apr. 24, '09) Associate Professor of Mining Engineering, University of Idaho, Moscow, Idaho.
CHIARAVIGLIO, Dino (Apr. 3, '02) Corso Vittorio Emanuelo 94, Turin, Italy.
CHILDS, D. H. (Apr. 3, '02) Alfred Univ., Alfred, N. Y.
CHORPENING, G. B. (Apr. 2, '04) Elec. Eng., Clarksburg, W. Va.
CITO, Camilo C. (Sept. 26, '08) Supt. Glorieux Smelt. & Ref. Co., Irvington, N. J.
CLAFLIN, Alan A. (Sept. 4, '03) Pres. Avery Chem. Co., 7 Sears St., Boston, Mass.
CLAMER, G. H. (Apr. 3, '02) Ajax Metal Co., Frankford Ave. and Richmond St., Philadelphia, Pa.
CLAPP, E. H. (Sept. 4, '03) Vice-Pres. Penobscot Chem. Fibre Co., 49 Federal St.; res., 490 Beacon St., Boston, Mass.
CLARK, Friend E., Ph.D. (Apr. 3, '02) Prof. of Chem., Central Univ. of Kentucky, Danville, Ky.

CLARK, Walter G. (Sept. 28, '07) Pres. Parker-Clark Electric Co., 135 Broadway, New York City.
CLARK, Wm. J. (Apr. 3, '02) Genl. Mgr. Foreign Dept., Genl. Elec. Co., 44 Broad St., New York City
CLARKE, William A. (May 29, '09) Treasurer, The Maclaren & Sprague Lbr. Co., Toledo, Ohio; mailing address, 1638 Broadway.
CLARKE, Eben B. (June 25, '09) General Manager, Firth-Sterling Steel Co., McKeesport, Pa.
CLEAVES, Dr. Margaret A. (Mar. 5, '04) Physician in active practice, 616 Madison Ave., New York City.
CLEVENGER, G. H. (Aug. 27, '09) Consulting Engineer, Butters Salvador Mines; mailing address, 381 Hawthorne Ave., Palo Alto, Cal.
CLYMER, W. R. (May 30, '08) Supt. National Carbon Co.; mailing address, 1426 West 107th St., Cleveland, Ohio.
COATES, Jesse (May 29, '09) Genl. Sales Mgr., Coatesville, Pa.
COGGESHALL, G. W. (Apr. 3, '02) 17 Chestnut St., Dedham, Mass.
COHO, H. B. (Apr. 3, '02) 26 Cortlandt St., New York City.
COHOE, Wallace P. (Oct. 28, '09) President, Chemical Laboratories, Ltd., 148 Van Horne St., Toronto, Canada.
COIT, Charles W. (Apr. 3, '02) 520 Culver Road, Rochester, N. Y.
COLBY, Ed. A. (Apr. 3, '02) Baker Platinum Works, Newark, N. J.
COLCORD, Frank F. (Oct. 7, '05) Chemist, U. S. Metals Ref. Co., Chrome, N. J.
COLE, Edward R. (June 25, '09) Assistant Superintendent, International Acheson Graphite Co., Niagara Falls, N. Y.
COLE, Henry N. (Mar. 27, '09)) Instructor in Chemistry University of Michigan, Ann Arbor, Mich.; mailing address, 702 Forest Ave.
COLEMAN, E. P. (May 29, '09) Steam Engineer, Lackawanna Steel Co., Buffalo, N. Y.; mailing address, 271 Franklin St.
COLLENS, C. L., 2d (Apr. 3, '02) Pres. Lincoln Motor Works Co., Room 721, Caxton Bldg., Cleveland, Ohio; res., 1933 E. 71st St.
COLLINS, Fred L. (Oct. 29, '08) Chief Electrician, Universal Portland Cement Co., Buffington, Ind.; res., 6020 Normal Ave., Chicago, Ill.
COMSTOCK, Louis K. (Sept. 26, '08) L. K. Comstock & Co., 195 Fernwood Ave., Upper Montclair, N. J.
CONGER, R. T. (Sept. 4, '03) Chemist, Chicago Edison Co., Chicago, Ill.
CONLIN, Frederick (Jan. 8, '04) 835 Kensington Ave., Plainfield, N. J.
CONVERSE, Vernon G. (Oct. 29, '08) Eng. in charge, Ontario Power Co.; P. O. Box 3, Niagara Falls, N. Y.
CONVERSE, W. A. (Nov. 27, '09) Secretary and Chemical Director, Dearborn Drug and Chemical Co., Chicago, Ill.; mailing address, 4320 Greenwood Ave.
COOPER, K. F. (Feb. 27, '09) Superintendent, American Cyanamid Co., Niagara Falls, Canada (Drawer 29).
CORIN, William (Dec. 26, '08) Electrical Engineer, Public Works Dept., Sydney, N. S. W., Australia.
CORNELIUS, Erik (Oct. 29, '08) Mgr. Zinc Works of A. B. Saxeberget, Trollhattan, Sweden.
CORNTHWAITE, S. (Sept. 25, '09) Manager, Electric Steel Works, Jossingford, Dalene, Norway.
CORSON, Wm. R. C. (Sept. 4, '03) Consult. Eng., 36 Pearl St., Hartford, Conn.
COWLES, Alfred H. (Apr. 3, '02) 361 The Arcade; res., 656 Prospect St., Cleveland, Ohio.
COWPER-COLES, S. (Oct. 10, '03) Grosvenor Mansions, Victoria St., London, Eng.
COX, G. E. (Apr. 3, '02) Supt. Union Carbide Works; res., 315 Buffalo Ave., Niagara Falls, N. Y.
CRABTREE, Prof. Fred. (May 29, '09) Prof. of Metallurgy and Mining, Carnegie Technical Schools, Pittsburgh, Pa.
CRANE, F. D. (Oct. 29, '08) Research Chemist, Synfleur Sci. Labs., Monticello, N. Y.; mailing address, 28 Hillside Ave., Montclair, N. J.
CREAGH, Edric C. (Sept. 26, '08) P. O. Box 383, Dunedin, New Zealand.
CREIGHTON, Elmer E. F. (Apr. 3, '02) South College, Union College, Schenectady, N. Y.
CRIDER, J. S. (May 9, '03) Sec. National Carbon Co., Lock Drawer "L," Cleveland, Ohio.
CRIDLAND, H. C. (Apr. 24, '09) Chemist and Metallurgist, The Joyce-Cridland Co., Dayton, Ohio; mailing address, 36 Potomac St.
CRIM, Lemuel P. (Jan. 29, '09) Draughtsman, U. S. Ordnance Dept.; mailing address, 4541 9th Ave., Seattle, Wash.
CROCKER, Dr. F. B. (Nov. 6, '02) Prof. of Elec. Eng., Columbia Univ.; res., 14 W. 45th St., New York.
CRUMBIE, Wm. D. (Nov. 26, '07) Chemist, U. S. Appraiser's Dept., New York City; mailing address, 146 Washington St., E. Orange, N. J.
CUSHMAN, Allerton S. (June 1, '07) Chemist, U. S. Dept. Agriculture; res., 1751 N St., Washington, D. C.
DAFT, Leo (Mar. 27, '09) Consulting Electrical Engineer, Rutherford, N. J.
DALBURG, Frank A. (May 29, '09) Surveyor, Bureau of Lands, Manila, P. I.

DANFORTH, Chas. W. (May 29, '09) Chief Chemist, Sharon Steel Hoop Co., Sharon, Pa.
DAPPLES, Alfred (Oct. 29, '08) Bussi-Afficienne, Aguila, Italy.
DAVIES, M. L. (Sept. 4, '03) Sec. and Treas. The North Amer. Chem. Co., Bay City, Mich.
DAVIS, Chas. H. (Feb. 27, '09) Pres., American Road Machine Co., South Yarmouth, Mass.
DAVIS, D. L. (Aug. 7, '02) Supt. The Salem Elec. L. and P. Co.; res., 299 Lincoln Ave., Salem, Ohio.
DAVIS, Robert W., Jr. (Feb. 27, '09) Chief Chemist, Haskell Plant, E. I. du Pont Powder Co., Haskell, N. J.
DAVIS, Stewart A. (Jan. 29, '09) Vice-President, Amer. Sheet Steel & Tin Plate Co., Frick Bldg., Pittsburgh, Pa.
DAVIS, Wm. H. (Sept. 17, '03) Metallurgist and Mill Supt., Idaho Gold Coin M. and M. Co.; res., 1720 Spruce St., Boulder, Col.
DECKER, Frank A. (Oct. 6, '06) Gen. Mgr. Decker Elec. Mfg. Co., 2011 Bellevue St.; mailing address, 1702 Ontario St., Philadelphia, Pa.
DEEDS, E. A. (Nov. 6, '02) Asst. Gen. Mgr. National Cash Register Co., 319 Central Ave., Dayton, Ohio.
DeNEUFVILLE, Dr. R. (Feb. 5, '03) Junghofstrasse 14, Frankfurt a|M, Germany.
DENNIS, Prof. L. M. (Sept. 4, '03) Cornell Univ., Ithaca, N. Y.
DENNISON, C. H. (Feb. 6, '04) Chemist, Revere Rubber Co., 8½ John St., Chelsea, Mass.
DEVENTER VAN, Harry R. (Feb. 27, '09) Elec. Engr., Sumter Tel. Mfg. Co., Sumter, S. C.
DEVEREUX, Washington (Jan. 6, '06) Inspector, Phila. Fire Underwriters' Association, 1625 N. 29th St., Philadelphia, Pa.
DEVLIN, S. B. (Jan. 8, '03) Chief Asst. and Dir. of Lab. of Dr. Paget, 156 W. 13th St.; res., 359 Seventh Ave., New York City.
DEWEY, F. P. (Apr. 2, '04) Assayer to the Mint Bureau, Lanier Heights, Washington, D. C.
DIAZ-OSSA, Prof. Belisario (June 25, '09) Prof. of Technology, University of Chile, Casilla No. 962, Santiago, Chile, South America.
DICKINSON, William N., Jr. (Sept. 26, '08) Mgr. Foreign Dept., Otis Elevator Co., 17 Battery Place, New York City.
DICKSON, Archibald A. C. (Mar. 27, '09) Consult. Engr., East India Railway Co., Rejouli P. O., via Newadah, Gaya District, India.
DILL, Colby (Nov. 27, '09) Works Mgr., Perth Amboy Chem. Works, Perth Amboy, N. J.; mailing address, 233 Water St.
DOERFLINGER, Wm. F. (July 3, '02) Consult. Eng., 52 Beaver St., New York City.
DOLT, Dr. Maurice L. (Apr. 30, '08) Instructor in Chemistry, Lehigh University, South Bethlehem; mailing address, Leonard Hall, Delaware Ave.
DOOLITTLE, C. E. (May 9, '03) Mgr. and Elec. Eng., The Roaring Fork Elec. Light and Power Co., Aspen, Col.
DOREMUS, Dr. Chas. A. (Apr. 3, '02) 55 W. 53d St., New York.
DORNEY, James J. (Sept. 26, '08) West El. & Mfg. Co., 605 Bank of Commerce Bldg., St. Louis, Mo.
DOTY, Ernest L. (Sept. 26, '08) Dist. Eng., Westinghouse El. & Mfg. Co., 784 Ellicott Square, Buffalo, N. Y.
DOUGHERTY, John W. (Feb. 27, '09) Genl. Superintendent, Pennsylvania Steel Co., Steelton, Pa.
DOUGLAS, J. (Nov. 27, '09) The Phelps-Dodge Co., 99 John St., New York City,
DOW, Herbert H. (Apr. 3, '02) Midland, Mich.
DOWNES, Arthur C. (July 30, '09) Chemist, National Carbon Co., Fostoria, Ohio.
DRAEGER, A. Bernhard (Nov. 26, '07) Draeger Werke, Lubeck, Germany.
DRAKE, Francis E. (June 6, '03) Representative La Societe Anonyme Westinghouse, 45 Rue de l'Arcade, Paris, France.
DREYFUS, Dr. Wm. (Dec. 4, '02) Chem. and Mgr West Disinfecting Co., 57 E. 96th St., New York.
DROBEGG, Dr. Gustave (Jan. 8, '03) 11 Bartlett St., Brooklyn, N. Y.; New York office, 81 Maiden Lane.
DRYER, Ervin (Sept. 4, '03) Salesman and Eng., W. E. and Mfg. Co.; res., 26 Ogden Ave., Chicago, Ill.
DUDLEY, Prof. Wm. L. (Jan. 8, '04) Prof. of Chemistry, Vanderbilt Univ., Nashville, Tenn.
DuFAUR, B. (June 1, '07) Assayer, Mt. Morgan, Queensland, Australia.
DUNCAN, Dr. Louis (Sept. 4, '03) Consult. Elec. Eng., 56 Pine St., New York.
DUNCAN, Thos. (Nov. 6, '03) Vice-Pres. and Gen. Mgr., Duncan Elec. Mfg. Co., 317 S. 6th St., La Fayette, Ind.
DUNLAP, Orrin E. (July 31, '08) Sect'y Int. Acheson Graphite Co., Niagara Falls, N. Y.
DUNN, Drew H. (Feb. 27, '09) Consult. Eng. and Geologist, 424-5 Bradbury Bldg., Los Angeles, Cal.
DU PONT, Irenee (Apr. 24, '09) E. I. Du Pont de Nemours Powder Co., Wilmington, Del.

DU PONT, Pierre S. (Jan. 29, '09) Treas., E. I. Du Pont de Nemours Powder Co., Wilmington, Del.
DURANT, Edw. (Apr. 3, '02) 115 E. 26th St., New York.
DUSCHAK, L. H. (June 25, '09) Physical Chemist, Research Laboratory, Corning Glass Works, Corning, N. Y. (Box 692).
DUSHMAN, Saul (June 25, '09) Lecturer in Electrochemistry, University of Toronto, Toronto, Canada; mailing address, 7 Havelock St.
EDGE, Dexter (Oct. 29, '08) Shenandoah Club, Swissvale, Pa.
EDISON, Thos. A. (Apr. 4, '03) Orange, N. J.
EDMANDS, I. R. (Aug. 7, '02) Union Carbide Co., 79 Wall St., New York City.
EDSTROM, J. Sigfrid (Nov. 5, '04) Managing Dir., General Electric Co. of Sweden, Vesteras, Sweden.
EGLIN, Wm. C. L. (July 1, '04) Elec. Eng., 235 S. 42d St., Philadelphia, Pa.
EIMER, A. (Dec. 4, '02) Eimer & Amend, 205-211 Third Ave., New York.
ELLIOTT, A. H. (Apr. 3, '02) Cons. Gas Co., 4 Irving Place, New York.
ELY, Theodore N. (Apr. 3, '02) P. R. R. Co., Chief of Motive Power, Broad St. Station, Philadelphia, Pa.
EMANUEL, Louis V. (Oct. 17, '07) Metallurgist, Amer. Smelt. and Ref. Co.; mailing address, 55 Rector St., Perth Amboy, N. J.
EMERY, A. L. (Apr. 3, '02) Smith, Emery & Co., Chem. and Met. Engs., Howard and Hawthorne Sts., San Francisco, Cal.
EMERY, W. L. (Sept. 26, '08) Foreman of Meter Dept., Utah Lt. & Ry. Co.; mailing address, 830 Emeril Ave., Salt Lake City, Utah.
EMRICH, H. H. (May 7, '04) Supt. copper refinery, Perth Amboy plant, A. S. & R. Co., Maurer, N. J.; res., 142 Water St., Perth Amboy, N. J.
ENGELHARD, Chas. (May 29, '09) President, American Platinum Works, Newark, N. J.; mailing address, Hudson Terminal Bldg., 30 Church St., New York City.
ENGELHARDT, Victor (Dec. 4, '02) Charlottenburg, Schlossstrasse 11. 2, Germany.
ENGLE, Horace M. (Aug. 31, '07) The Southern Development Co., Terry Bldg., Roanoke, Va.
ERHART, W. H. (Dec. 27, '07) 11 Bartlett St., Brooklyn, N. Y.
EURICH, E. F. (Nov. 27, '09) Mining and Metallurgical Engineer, 15 William St., New York City; res., Montclair, N. J.
EVANS, Chas. (Oct. 28, '09) care of Carter & Scattergood, Riverton, N. J.
EVANS, H. S. (Apr. 3, '02) Prof. Elec. Eng., Univ. of Col., Boulder, Col.
EVANS, J. W. (Apr. 3, '02) Belleville, Ontario, Canada.
EVERETTE, Dr. Willis E. (July 30, '09) Consulting Chemical and Mining Engineer, Tacoma, Wash.
EWIN, Jas. L. (Nov. 6, '02) Patent Solicitor, 900 F St., N. W., Washington, D. C.
EWING, A. J. (Apr. 2, '04) 4 Fairfield St., Canton, Cardiff, Wales.
FAHY, J. T. (May 29, '09) Electrician, New Zealand Railways, Wellington, New Zealand.
FAHRIG, Ernst (Sept. 4, '03) 1017-18 Betz Bldg., Philadelphia, Pa.
FAIRCHILD, John G. (July 30, '09) Analytical Chemist, 1747 F St., Washington, D. C.
FALDING, F. J. (Apr. 3, '02) Consult. Eng., 55 Broadway, New York.
FAUST, Thomas B. (Jan. 29, '09) Genl. Superintendent, Red River Furnace Co., Clarksville, Tenn.
FAWCETT, Percy (Sept. 25, '09) Director, Thomas Firth & Sons, Ltd., Sheffield, England.
FERGUSON, William A. (June 25, '09) Electrical Engineer, Mexican Light & Power Co., Apartado 490, Mexico City, Mexico.
FERNBERGER, H. M. (Jan. 8, '04) Chemist, 1939 N. 12th St., Philadelphia, Pa.
FICHTER, Dr. Fritz (Nov. 26, '07) Prof. of Organic Chem., Univ. of Basel; res., 24 Marschalkenstrasse, Basel, Switzerland.
FINK, Dr. Colin G. (Nov. 26, '07) Research Chemist, General Electric Co., P. O. Box 719, Schenectady, N. Y.
FITZ GERALD, C. M. (May 30, '08) The Evening Sun, 170 Nassau St., New York City.
FITZGERALD, F. A. J. (Apr. 3, '02) FitzGerald & Bennie Laboratories, Niagara Falls, N. Y.
FITZGIBBON, R. (Apr. 3, '02) 534 Canal St., New York.
FLAGG, Stanley G., Jr. (Jan. 29, '09) Mgr., Stanley G. Flagg & Co., 424 N. 19th St., Philadelphia.
FLANIGAN, John T. (Nov. 27, '09) Consulting Chemist, Ever Ready Dry Battery Co., Paterson, N. J.; mailing address, 71 Marshall St.
FLEMING, R. (Apr. 3, '02) 148 Elmwood Road, Swampscott, Mass.
FLIESS, Robert A. (Sept. 4, '02) 13 S. Arlington Ave., E. Orange, N. J.
FLOWERS, Alan E. (Oct. 29, '08) Asst. Prof. of Elec. Engr., University of Missouri, Columbia, Mo.; mailing address, 26 Allen Place.
FOERSTERLING, Dr. Hans (Apr. 3, '02) Roessler & Hasslacher Chem. Co., Perth Amboy, N. J.

FOLK, Geo. E. (Sept. 25, '09) Patent Lawyer, Barton & Folk, 1445 Monadnock Bldg., Chicago, Ill.
FOOTE, Arthur DeWint (Feb. 27, '09) Superintendent, North Star Mines, Grass Valley, Cal.
FOREGGER, Richard von Ph.D. (Sept. 2, '05) care of Roessler & Hasslacher Chem. Co., 100 William St.; res., 48 E. 87th St., New York.
FORSSELL, J. (June 1, '07) Hoganas, Sweden.
FOSTER, Charles E. (July 31, '08) Mgr. Cambridge Dept., Taylor Inst. Co., Ames St., Rochester, N. Y.
FOSTER, Oscar R. (Apr. 30, '08) Chem. De La Vergne Mach. Co., 549 Monroe St., Brooklyn, N. Y.
FOWLER, R. E. (Nov. 6, '03) Chemist, The National Electrolytic Co., Niagara Falls, N. Y.
FOWLER, Samuel S. (Apr. 3, '02) Min. Eng., P. O. Drawer 1024, Nelson, B. C.
FRALEY, Jos. C. (Apr. 3, '02) Attorney-at-Law, 1815 Land Title Bldg.; res., 1833 Pine St., Philadelphia, Pa.
FRANK, KARL G. (Feb. 27, '09) Electrical Engr., Siemens & Halske Co., 50 Church St., New York City.
FRANKFORTER, Prof. G. B. (Apr. 3, '02) Univ. of Minnesota, Minneapolis, Minn.
FRANKLIN, Milton W. (May 29, '09) Engineer, P. & M. Eng. Dept., General Electric Co., Schenectady, N. Y.
FRANKLIN, Prof. W. S. (Mar. 4, '05) Professor of Physics, Lehigh Univ., South Bethlehem, Pa.
FRARY, Francis C. (Aug. 31, '07) Instructor in Chemistry, Univ. of Minnesota, Minneapolis, Minn.
FRASCH, Hans A. (May 9, '03) Consult. Chem. Eng., 52 Broadway, New York.
FREDERICK, Geo. E., Jr. (Jan. 29, '09) Mgr., Arnold Hoffman Co., P. O. Box 762, New York City.
FREE, Edward E. (Apr. 7, '06) Chemist, Bureau of Soils, Dept. of Agriculture, Washington, D. C.
FREEDMAN, Prof. W. H. (Apr. 3, '02) Prof. of Elec. Eng., Univ. of Vermont; res., 100 S. Union St., Burlington, Vt.
FREEMAN, Gay N. (July 30, '09) Assayer and Analytical Chemist, Thermopolis, Wyo.
FRENZEL, A. B. (May 9, '03) Proprietor of mines containing rare minerals, 512 Equitable Bldg., Denver, Col.
FRIEDLAENDER, Eugene (May 29, '09) Superintendent, Elec. Dept., Carnegie Steel Co.; mailing address, 101 Kirkpatrick Ave., Braddock, Pa.
FRIES, Harold H., Ph.D. (May 1, '06) 92 Reade St., New York.
FRITCHLE, Oliver P. (Sept. 4, '02) Chemist, 1453 Clarkson St., Denver, Col.
FRODSHAIN, Harold (Nov. 21, '08) Research Chemist, Gen. El. Co., P. O. Box 842, Schenectady, N. Y.
FURNESS, Radclyffe (May 29, '09) Engineer in Charge of Research, Midvale Steel Co., Philadelphia.
GABRIEL, Geo. A. (April 3, '02) 432 Westminster Road, Brooklyn, N. Y.
GAGE, Robert B. (Feb. 27, '09) Chief Chemist, Geological Survey of New Jersey, Survey Laboratory, Green's Alley, Trenton, N. J.
GAHL, Dr. Rudolph (June 6, '03) Morenci, Arizzona.
GAINES, Richard H. (Oct. 17, '07) New York Board of Water Supply, 147 Varick St., New York City.
GALL, Henry (Apr. 2, '04) Societe de Electrochimie, 2 Rue Blanche, Paris, France.
GAMBA, F. P. (Apr. 24. '09) Rector of the National School of Mines, Pasto, Columbia, S. A. (via Panama y Tumaco).
GANDILLON, Ami (Jan. 8, '04) Case 6219, Bourg de Four, Geneva, Switzerland.
GANZ, Albert F. (Nov. 21, '08) Prof. of Electrical Engineering, Stevens Institute of Technology, Hoboken, N. J.; res., 612 River St.
GARDNER, Henry A. (Oct. 28, '09) Director, Paint Manufacturers' Association of United States, 3500 Grays Ferry Road, Philadelphia.
GARFIELD, A. S. (Mar. 7, '03) 67 Ave. de Malakoff, Paris XVI, Arrond, France.
GEER, Wm. C. (Nov. 26, '07) Chief Chemist, B. F. Goodrich Co., Akron, Ohio; mailing address, 23 Hawthorne Ave.
GEIB, Wm. V. (Nov. 27, '09) Salesman, Westinghouse Electric and Mfg. Co., Baltimore, Md.; mailing address, 121 E, Baltimore St.; res., 1917 Linden Ave.
GERRY, M. H., Jr. (Apr. 3, '02) Gen. Mgr. and Chief Eng. Missouri River Power Co., Helena, Mont.
GIBBS, Arthur E. (Oct. 2, '02) Mfg. Chemist, Wyandotte, Mich.
GIBBS, W. T. (Apr. 3, '02) Director, The Electric Reduction Co., Ltd., Buckingham, Quebec, Canada.
GIFFORD, Wm. E. (Apr. 3, '02) 408 N. J. R. R. Ave., Newark, N. J.
GILCHRIST, Peter S. (Apr. 3, '02) Chem. Eng., Charlotte, N. C.
GILES, Delos C. (Oct. 17, '07) Grover, Bradford Co., Pa.
GILES, Irvin K. (May 1, '06) Niagara Electrochem. Co., Niagara Falls, N. Y.
GIN, Gustave (Dec. 4, '03) Ingenieur Electrometallurgiste, 149 Rue de Rome, Paris (XVII), France.

GIOLITTI, Federico, Ph.D. (Oct. 17, '07) Professor of Metallurgy, Regio Politecnico, Turin, Italy.
GIRDWOOD, Gilbert P., M.D. (May 29, '09) Emeritus Professor of Chemistry, McGill University, Montreal, Canada; mailing address, 111 University St.
GIRDWOOD, Kennet J. (Nov. 21, '08) Apartado 34, El Oro, Mexico.
GLADSON, Prof. W. N. (Apr. 3, '02) Elec. Eng. Dept. Univ. of Arkansas, 120 W. Maple St., Fayetteville, Ark.
GLENCK, I. A. H. (Oct. 10, '03) Consult. Eng., Gallus Anlage I, Frankford a|M, Germany.
GLUCROFT, Samuel H. (Dec. 26. '08) El. Eng., with Glucroft & Glucroft, 52 Graham Ave., Brooklyn, N. Y.
GODDARD, Chris. M. (Apr. 3, '02) Nat. Board of Fire Underwriters, 141 Milk St., Boston; res., 1008 Beacon St., Newton Centre, Mass.
GOEPEL, Carl P. (Nov. 4, '05) Patent-counsel, Goepel & Goepel, 290 Broadway; res., 2350 7th Ave., New York.
GOLDBAUM, Jacob S. (Nov. 27, '09) Instructor in Electrochemistry, Univ. of Pennsylvania, Philadelphia; res., 1745 N. 8th St.
GOLDSCHMIDT, Dr. Hans (Nov. 6, '03) Chem. Mfr., Essen-Ruhr, Germany.
GOODRICH, C. C. (Apr. 3, '02) Llewellyn Park, Orange, N. J.
GOODWIN, H. M., Ph.D. (Apr. 3, '02) Mass. Inst. of Tech., Boston, Mass.
GOODWIN, Jos. H. (Jan. 6, '06) Chemist, Western Electric Co., 151 N. 15th St. E. Orange, N. J.
GOODWIN, W. L., D.Sc. (Apr. 3, '02) Director, School of Mining, Kingston, Ont., Canada.
GORDON, Prof. C. McC. (Apr. 3, '02) Lafayette College, Easton, Pa.
GORDON, Henry A. (Apr. 24, '09) Consulting Engineer, Ben Lomon, Ranfirely Road, Epsom, Auckland, New Zealand.
GORMAN, Harry B. L. (Apr. 24, '09) Electrical Engineer, The Gisholt Machine Co., 214 Buell St., Madison, Wis.
GRADOLPH, William F. (Nov. 21, '08) Pres. and Chief Eng., Gradolph El. Co., 800 Chestnut St., St. Louis, Mo.; res., 2908 St. Vincent St.
GRAHAM, James (May 29, '09) Chemist, Acheson Oildag Co., Niagara Falls, N. Y.
GRANBERY, J. H. (Apr. 3, '02) Eng., Buel & Mitchell, 120 Liberty St., New York,
GRAVES, Carleton A. (Sept. 26, '08) Power Eng., 360 Pearl St., Brooklyn, N. Y.
GRAVES, Walter G. (Mar. 5. '03) Supt. Grasselli Chem. Co.; res., 1950 E. 90th St., Cleveland, Ohio.
GRAY, C. W. (Oct. 28, '09) Chief Chemist, Sinnamahoning Powder Mfg. Co., Sinnamahoning, Pa.
GREENE, Albert E. (Aug. 31, '07) Electro-Metallurgical Engineer, 1445 Monadnock Bldg., Chicago, Ill.
GREENE, F. V. (Nov. 27, '09) President, Niagara, Lockport & Ontario Power Co.; Vice-president, The Ontario Power Co. of Niagara Falls.; mailing address, Fidelity Bldg., Buffalo, N. Y.
GRIFFIN, Martin L. (Oct. 1, '04) The Emerson Lab., 177 State St., Springfield, Mass.
GROSVENOR, Dr. Wm. M. (June 1, '07) Chemical Engineer, 1123 Broadway, New York.
GROWER Geo. G. (Nov. 5, '04) Coe Brass Mfg. Co.; res., 48 Cottage Ave., Ansonia, Conn.
GUDEMAN, Edward (Feb. 27, '09) Consulting Chemist, 903-4 Postal Telegraph Bldg., Chicago, Ill.
GUERBER, Arnold J. (July 31, '08) 1328 Walnut St., Boulder, Colo.
GUESS, Geo. A. (Aug. 5, '05) Metallurgist, Tennessee Copper Co., Copper Hill, Tenn.
GUTTMANN, Dr. Leo Frank (Oct. 29, '08) Chemistry Dept., Queen's University, Kingston, Ontario, Canada.
GUYE, Prof. Philippe A. (Dec. 4, '02) 3 Chemin des Cottages, florissant, Geneve, Switzerland.
HAANEL, Dr. Eugene (July 31, '07) Director of Mines, Dept. of Mines, Ottawa, Can.
HAAS, Herbert (May 7, '04) 320 Market St., San Francisco, Cal.
HABER, Prof. Dr. F. (Nov. 6, '02) Technische Hochschule, Karlsruhe in Baden, Germany; res., Moltke Str. 31.
HADFIELD, R. A. (July 6, '06) Manag. Dir. Hadfield Steel Fdy. Co., Ltd, Parkhead House, Sheffield, England.
HADLEY, A. N. (Apr. 3, '02) Box 33, Indianapolis, Ind.
HAFF, Max M. (Aug. 7, '03) Research Electrochemist, care of Laboratory, 188 Metcalfe St., Ottawa, Canada.
HAGGOTT, Ernest A. (Apr. 3, '02) Engineer of Mines, 2525 Powell St., Los Angeles, Cal.
HALL, Chas. M. (Apr. 3, '02) Aluminum Co. of America, Niagara Falls, N. Y.
HALL, Samuel F. (Apr. 16, '03) Supt., Norton Co., Niagara Falls, N. Y.
HALLETT, Lucius F. (Sept. 26, '08) 900 Logan Ave., Denver, Col.
HAMBUECHEN, Carl. E. E. (Apr. 3, '02) Electrochemist, Madison, Wis.

HAMMER, Wm. J. (Aug. 7, '03) Consult. Elec. Eng., 26 Cortlandt St., and 153 W. 46th St., New York.
HAMMOND, John F. (June 1, '07) Designer of Electrical Apparatus, S. S. White Dental Mfg. Co., Prince Bay, S. I., New York.
HANKS, M. W. (Sept. 4, '03) Engineer, De Pere, Wis.
HANSEN, C. A. (May 29, '09) Research Laboratory, General Electric Co., Schenectady, N. Y.
HARPER, Dr. H. W. (Apr. 3, '02) Univ. of Texas, Austin, Texas.
HARPER, John L. (Apr. 6, '07) Chief Eng. Niagara Falls Hydr. Power and Mfg. Co.; res., 148 Buffalo Ave., Niagara Falls, N. Y.
HARRAR, Ellwood S. (Feb. 27, '09) Pittsburg & Conneaut Dock Co., Ashtabula, Ohio; res., 72 Station St.
HARRINGTON, Dr. E. I. (Apr. 3, '02) 84 Ashburton Ave., Yonkers, N. Y.
HARRIS, J. W. (Apr. 3, '02) U. S. Patent Office, Washington, D. C.
HARRIS, Jonathan W. (Sept. 26, '08) Research Chemist, Western El. Co., 463 West St., New York City.
HARRISON, Herbert C. (May 29, '09) Vice-President, Susquehanna Smelting Co., Ashleigh Bldgs., Lockport, N. Y.
HART, Carl D. (Apr. 24, '09) Engineer, Western Electric Co., New York City; mailing address, 37 W. 129th St.
HART, Ed., Ph.D. (Aug. 7, '02) Prof. of Chem., Lafayette College, Easton, Pa.
HART, L. O. (Nov. 27, '09) Electrical Engineer, Driver Harris Wire Co., Harrison, N. J.; res., 232 Washington St., Hoboken, N. J.
HARTLEY, Robt. H. (Dec. 27, '07) Chemist, Hartley Bldg., Fourth Ave. and Smithfield Sts., Pittsburgh, Pa.
HARVEY, E. F. (Apr. 3, '02) St. John's, Newfoundland.
HASKELL, F. W. (Apr. 3, '02) Pres. Carborundum Co., Niagara Falls, N. Y.
HASLWANTER, Chas. (April 3, '02) 447 Spruce St., Richmond Hill, L. I., N. Y.
HASSLACHER, Jacob (Nov. 26, '07) Pres. Roessler & Hasslacher Chem. Co., 100 William St.; P. O. Box 1999, New York City.
HATCH, Israel (Oct. 28, '09) Asst. Superintendent, Elgin National Watch Co., Elgin, Ill.
HATZEL, J. C. (Apr. 3, '02) 571 Fifth Ave.; res., 89 W. 119th St., New York.
HAUSER, S. T. (Mar. 25, '08) Sect'y Missouri River Power Co., Helena, Mont.
HAY, John A. (May 29, '09) care of Grondal-Kjellin Co., 45 Wall St., New York City.
HAYES, Clifton R. (Sept. 26, '08) Supt. El. Dept., Fitchburg Gas & El. Lt. Co., Fitchburg, Mass.
HEARNE, D. Garth (Jan. 29, '09) President, Eagle Fluor Spar Co., Wheeling, W. Va.
HEATH, H. E. (Apr. 3, '02) Engineer, Gen. Elec. Co.; res.. 99 Laighton St., Lynn, Mass.
HEDLUND, Marten (Apr. 1, '05) Gullspangs Elektrochemishe Aktenbolog, Gullspang, Sweden.
HEITMANN, Edward (Sept. 26, '08) El. Eng., Crocker Wheeler Co., Ampere, N. J.; res., Montclair, N. J.
HEIZMANN, L. J. (Oct. 29, '08) Asst. Treas. and Chief Eng., Penn Hardware Co.,; res. 318 N. Fifth St., Reading, Pa.
HEMINGWAY, Frank (Nov. 26, '07) Mfg. Chemist, with Hemingway & Co., 133 Front St., New York; res., 131 Grove St., Montclair, N. J.
HENDRY, W. Ferris (Nov. 26, '07) Western El. Co., 463 West St., New York City.
HENNIG, Charles T. (April 24, '09) Consulting Chemist, 1620 New York Ave., Brooklyn, N. Y.
HENNING, Clarence I. B. (May 7, '04) care E. I. duPont de Nemours Powder Co., Haskell, N. J.
HEPBURN, Donald MacKnight (May 29, '09) Genl. Manager, Niagara Lead Co., Niagara Falls, N. Y.
HERAEUS, Heinrich (Nov. 6, '03) Hanau, Germany.
HERING, Carl (Apr. 3, '02) Consult. Eng., 929 Chestnut St., Philadelphia, Pa.
HEROULT, Dr. Ing. P. L. T. (Jan. 8, '04) The Ansonia, 73d St. and Broadway, New York.
HERZOG, F. Benedict, Ph.D. (Apr. 3, '02) Herzog Teleseme Co., 51 W. 24th St., New York.
HERZOG, G. K. (Feb. 27, '09) The Aluminum Co., of America, Massena, N. Y.
HESS, Frederick M. (Nov. 27, '09) President and General Manager, Inyo Telephone Co., Bishop, Cal.
HIBBARD, Henry D. (May 30, '08) Consult. Eng., 144 E. Seventh St., Plainfield, N. J.
HIGGINS, Aldus C. (Sept. 4, '02) Norton Co., Worcester, Mass.
HILL Nicholas S., Jr. (Nov. 27, '09) 100 William St., New York City; res., 381 William St., East Orange, N. J.
HILL, S. T. H. (Feb. 2, '07) Electrochemist of the Helena and Livingstone Smelting and Reduction Co.; res., 810 Benton Ave., Helena, Mont.
HILLIARD, John D. (Sept. 26, '08) El. Eng., Hudson River El. P. Co.; res., 103 Columbia St., Albany, N. Y.
HINRICHS, Dr. Gustavus (May 29, '09) Chemical Expert, 4106 Shenandoah Ave., St. Louis, Mo.

HIORTH, Frederick V. L. (Dec. 26, '07) Electrochem. Eng., Josefinegade 19, I, Christiania, Norway.
HIRSCH, Alcan (June 29, '07) Research Lab., Room 51, Pierce Bldg., Mass. Inst. of Tech., Boston, Mass.
HIRSCHLAND, Franz H., Dr. Ing. (Dec. 27, '07) Mgr. Goldschmidt Chem. Co., 60 Wall St., New York City.
HITCHCOCK, Fanny R. M. (May 1, '06) 4038 Walnut St., Philadelphia, Pa.
HITCHCOCK, Prof. G. G. (July 1, '04) Prof. of Physics, Pomona College, Claremont, Cal.
HITCHCOCK, Halbert K. (Oct. 2, '02) Experimental Eng., Tarentum, Pa.
HITE, B. H. (Apr. 4, '03) W. Va. Exp. Station, Morgantown, W. Va.
HITT, Samuel E. (Mar. 27, '09) Draftsman and Designer, Lackawanna Steel Co., 426 W. 3d St., Elyria, Ohio.
HOBBLE, Arthur C. (Nov. 6, '03) Elec. Eng., Sivassamudram, Mysore Province, South India.
HOBBS, Perry L., Ph.D. (Apr. 3, '02) Cowell Port. Cement Co., Baypoint, Cal.
HOFFMAN, Ottokar (Apr. 3, '02) 2110 Troost Ave., Kansas City, Mo.
HOFFMAN, Rudolf (Dec. 26, '08) Prof. of Metallurgy, Kgl. Bergakademie, Clausthal, Germany; mailing address, 221 Bergstrasse.
HOGEL, Hascal A, (Dec. 26, '08) 144 W. 16th St., New York City.
HOLLAND, Walter E. (Aug. 5, '05) Foreman, Exp. Testing Dept., Edison Storage Bat. Co.; res., 211 Arlington Ave., E. Orange, N. J.
HOLSTEIN, Otto (Nov. 21, '08) Gen. Foreman of Telegraph, Cerro de Pasco Ry. Co.; mailing address, Jefe, General de Telegrafes, La Oroya, Peru, S. A.
HOLTON, Fred. A. (Apr. 2, '04) Chemist in Patent Causes, 620 F St., N. W., Washington, D. C.
HONEY, Wm. (May 1, '07) Engineer in charge, Gen. Station., Tequisquiapan, Queretaro, Mexico.
HOOKER, Albert H. (Feb. 27, '09) Manager and Chemist, The Development & Funding Co., Niagara Falls, N. Y.
HORNOR, H. A. (Feb. 27, '09) Electrical Engr., The New York Shipbuilding Co., Camden, N. J.; mailing address, Hamilton Court, Philadelphia.
HORRY, Wm. S. (Feb. 5, '03) Cons. Lake Sup. Power Co., Niagara Falls, N. Y.
HOSKINS, Wm. (Apr. 3, '02) 81 S. Clark St., Chicago; res., La Grange, Ill.
HOUGH, Arthur (May 1, '06) The Detonite Explosives, Ltd., 400 St. James St., Montreal, Canada.
HOWARD, Geo. M. (Apr. 3, '02) Elec. Stor. Bat. Co., 19th St. and Allegheny Ave., Philadelphia, Pa.
HOWARD, Henry (Apr. 3, '02) 36 Amory St., Brookline, Mass.
HOWARD, L. E. (May 29, '09) Experimental Engineer, Simons Mfg. Co., 2418 West 16th St., Chicago.
HOWARD, Prof. S. F. (Apr. 3, '02) Dept. of Chem., Mass. Agricultural College; res., 10 Allen St., Amherst, Mass.
HOWE, Henry M. (Aug. 7, '02) Prof. of Metallurgy, Columbla University, New York City; res., McLean Road, Bedford Station, N. Y.
HOWELL, Wilson S. (Sept. 4, '03) 80th St. and East End Ave., New York.
HUDSON, Arthur J. (Nov. 26, '07) Patent Attorney, with Bates, Fonts & Hull, Society for Savings Bldg., Cleveland, Ohio.
HUFFARD, Jno. B. (Sept. 25, '09) Metallurgist, Electro-Metallurgical Co., Glen Ferris, W. Va.
HULETT, Geo. A. (Apr. 2, '04) 2 Murray Place, Princeton, N. J.
HUMBERT, Ernest P. (Mar. 27, '09) Electro-Metals Co., Welland, Ontario, Canada.
HUMPHRIES, Chad H. (Apr. 24, '09) Chemical Engineer, Westinghouse Lamp Co., Watsessing (near Bloomfield), N. J.; mailing address, 284 Kearny Ave., Kearny, N. J.
HUNGERFORD, Oliver T. (Jan. 29, '09) General Manager, Dielectric Co. of Amer., Belleville, N. J.
HUNT, A. M. (Apr. 3, '02) Consult. Eng., Union Trust Bldg., San Francisco, Cal.
HUTCHINGS, James T. (Sept. 26, '08) Asst. Gen. Mgr., Rochester Ry. & Lt. Co., Rochester, N. Y.
HUTCHINSON, E. J. (Apr. 3, '02) Vice-Pres. Taylor Chem. Co., 1245 E. 3d St., Cincinnati, Ohio.
HUTTON. R. S., D.Sc. (Apr. 3, '02) West St., Sheffield, England.
HYDE, Edward P. (Oct. 29, '08) Director, Physical Lab., National El. Lamp Assoc., Cleveland, Ohio.
IHLDER, John D. (April 3, '02) Elec. Eng., 17 Battery Place, New York City.
INGALLS, Walter R. (June 29, '07) 94 Bement Ave., West New Brighton, S. I., N. Y.
IRGENS, Johann F. (Jan. 28, '08) El. Eng. and Mgr., Technisk Bureau, Ltd., Walkendorffsgade 12, Bergen, Norway.
IRVINE, H. A. (April 3, '02) 142 Argyle Road, West Ealing, England.
ISAACS, A. S. (Apr. 3, '02) 404 Smithfield St., Pittsburgh, Pa.
ISAKOVICS, Alois von (Apr. 3, '02) Proprietor Synfleur Scientific Laboratories, Monticello, N. Y.
JACKSON, Prof. Dugald C. (Apr. 3, '02) Mass. Inst. Tech., Boston, Mass.

JACKSON, Alf. Geo. (July 30, '09) Manager, Synchronome Electric Co., of Australia, Ltd.; res., 65 Ann St., Brisbane, Queensland, Australia.
JAMES, Dr. J. H. (Apr. 3, '02) Chem. Dept., Carnegie Technical School, Pittsburgh, Pa.
JAMES, Rees (May 29, '09) Supt., Open Hearth Dept., Carnegie Steel Co., Munhall, Pa.
JENKINS C. Francis (Mar. 27, '09) Mech. Eng., Single Service Pkg. Corp., 1808 Park Rd., Washington, D. C.
JENKINS, DAVID J. (May 29, '09) Electrical Engineer, The Semet-Solvay Process Co., Syracuse, N. Y.; mailing address, 220 Center St., Milton, Pa.
JENKS, W. J. (Apr. 3, '02) 120 Broadway, New York.
JENNINGS, Edward P. (Feb. 27, '09) Consulting Engineer, Salt Lake City, Utah.
JENNISON, Herbert C. (Feb. 28, '08) Test. Lab., Coe Brass Mfg. Co., Box 313, Ansonia, Conn.
JEPPSON, Geo. N. (Sept. 4, '02) Norton Co., Worcester, Mass.
JOHANSEN, George H. (Aug. 27, '09) Civil Engineer, Box 94, Vinderen, Christiania, Norway.
JOHNS, John (Feb. 27, '09) Chief Assayer, Baltimore Copper S. & R. Co., Baltimore, Md.; res., 3042 O'Donnell St.
JOHNS, Morgan, J. (June 25, '09) Constructional Draftsman, The Electrolytic Co. of Australia, Port Kembla, N. S. W., Australia.
JOHNSON, Arden R. (June 2, '06) 244 E. Webster Ave., Chicago, Ill.
JOHNSON, M. H. (Nov. 27, '09) President and Treasurer, J. & M. Elec. Co., 94 Genesee St., Utica, N. Y.; res., 41 Plant St.
JOHNSON, Prof. Otis C. (Nov. 6, '03) 730 Thayer St., Ann Arbor, Mich.
JOHNSON, Woolsey McA. (Apr. 3, '02) 69 Vernon St., Hartford, Conn.
JOHNSTON, Frederick A. (Apr. 24, '09) Supt., Assay and Metallurgical Division, The S. S. White Dental Mfg. Co., Prince Bay, N. Y.
JOHNSTON, Wm. A. (Oct. 10, '03) Supt. of Mfg., S. S. White Dental Mfg. Co., Prince Bay, S. I., N. Y.
JONES, Harold (May 29, '09) Metallurgist, The St. John del Rey Gold Mining Co., Villa Nova de Lima, Minas Geraes, Brazil.
JONES, L. J. W. (Apr. 3, '02) c|o Secretary.
JOYCE, Clarence M. (May 1, '06) Chem., The Arlington Co., Box 23, Arlington, N. J.
KAHLENBERG, Louis, Ph.D. (Apr. 3, '02) 234 Lathrop St.; Prof. of Phys. Chem., Univ. of Wisconsin, Madison, Wis.
KAHN, Dr. Julius (May 1, '06) 161 Lafayette St., New York.
KAMMERER, Jacob A. (Oct. 28, '09) President and Gen Mgr., Wood Products Co. of Canada, Toronto, Canada; res., 87 Jameson Ave.
KATSURA, Prof. Benzo (May 29, '09) Prof. of Metallurgy, The Imperial Tokio University Tokio, Japan. (Temporary address, 60 Wall St., New York City, care of Takata & Co.)
KEBBE, George M. (July 31, '08) Asst. Gen. Agt., Edison Ill. Co., 360 Pearl St., Brooklyn, N. Y.
KEFFER, Frederic (Feb. 27, '09) Consult. Engr. and Geologist, The British Columbia Copper Co., Greenwood, B. C., Canada.
KEITH, Dr. N. S. (April 3, '02) 350 Bullitt Bldg., Philadelphia, Pa.
KELLER, Ed. (Apr. 3, '02) Anaconda Laboratory, Perth Amboy, N. J.
KELLER, Ch. A. (June 25, '09) Genl. Mgr., Keller-Leleux Cie., 3 Rue Vignon, Paris, France.
KELLY, Dr. John F. (Apr. 3, '02) Stanley Elec. Mfg. Co.; res., 284 W. Housatonic St., Pittsfield, Mass.
KELLOGG, Harry W (Jan. 29, '09) Genl. Mgr., National Electrolytic Co., Niagara Falls, N. Y.
KEMERY, Philo (May 29, '09) Metallurgical Engineer, Crescent Works, Crucible Steel Co. of America, Pittsburgh, Pa.
KENAN, Wm. R., Jr. (Apr. 3, '02) 242 Genessee St., Lockport, N. Y.
KENDALL, George R. (Jan. 28, '08) Lecturer in Chem., McGill Univ. College; res., 1629 Comox St., Vancouver, B. C., Canada.
KENNEDY, J. J. (May 9, '03) Engineer, 52 Broadway, New York.
KENNELLY, Dr. Arthur E. (Nov. 6, '02) Prof. of Elec. Eng., Pierce Hall, Harvard University, Cambridge, Mass.
KENT, Jas. M. (Sept. 4, '03) Teacher of Applied Steam and Electricity, Manual Training High School, 2726 Holmes St., Kansas City, Mo.
KENYON, O. A. (Nov. 27, '09) Editor, McGraw-Hill Book Co., 239 W. 39th St., New York City; res., 561 W. 186th St.
KENYON, Wm. H. (Apr. 3, '02) 49 Wall St., New York.
KERN, Ed. F. (Apr. 4, '03) care of Dept. of Metallurgy, Columbia Univ., New York.
KERR, Chas. H. (Mar. 2, '07) Research Chemist, Am. Optical Co., Southbridge, Mass.
KIER, Samuel M. (Oct. 29, '08) Pres. Kier Firebrick Co., 741 Sixth Ave., Pittsburgh, Pa.
KING, Robert O. (Apr. 7, '04) Elec. Eng., N. Tonawanda, N. Y.

KING, John E. (Feb. 27, '09) Principal, Big Springs High School, Big Springs, Texas.
KIRCHOFF, Chas. (Jan. 29, '09) 422 West End Ave., New York City.
KIRKEGAARD, Peter (Feb. 27, '09) Mgr., Deloro Mining & Reduction Co., Deloro, Ontario, Canada.
KISHI, Keijiro (Nov. 21, '08) Chief Eng., El. Dept., Shibaura Eng. Wks., No. 1, Shinhanacho, Kanasugi, Shibaku, Tokyo, Japan.
KITSEE, Dr. Isador (Apr. 3, '02) 306 Stock Exchange Place, Philadelphia, Pa.
KJELLIN, F. A. (Feb. 4, '05) Engineer for Elec. Melting, Metallurgiska Patent Aktiebolag, Stockholm, Sweden; res., Sturegatan 56, Stockholm, Sweden.
KLIPSTEIN, Ernest C. (Apr. 3, '02) 122 Pearl St., P. O. Box 2833, New York; res., 116 Prospect St., East Orange, N. J.
KNAPP, Geo. O. (Nov. 6, '02) Vice-Pres. Union Carbide Co., 157 Michigan Ave., Chicago, Ill.
KNEY, Otto (Sept. 26, '08) Adv. Mgr., Northern El. Co., Box 413, Madison, Wis.
KOCH, Stanley B. (May 29, '09) Chemist, New Jersey Zinc Co. of Pa., Horse Head Inn, Palmerton, Pa.
KOEHLER, Wm. (Nov. 5, '04) Consult. Electrochem. Eng., 4706 Superior St., Cleveland, Ohio.
KOHLER, Hy. L. (Aug. 31, '07) Chemist, Scullion Galliher Iron and Steel Co.; res., 3333A Nebraska Ave., St. Louis, Mo.
KOHN, Milton M. (May 29, '09) Eastern Sales Mgr., The Hoskins Mfg. Co., 253 Broadway, New York City.
KOWALKE, O. L. (Aug. 3, '06) Asst. in Applied Chem. and Research Work, Chem. Eng. Bldg., Madison, Wis.
KREJEI, Milo W. (Mar. 27, '09) Boston & Mont. C. C. & S. M. Co., 1200 6th Ave., N., Great Falls, Mont.
KREMERS, John G. (July 31, '07) Genl. Supt. Wisconsin Sugar Co., Menominee Falls; res., 554 Fourth St., Milwaukee, Wis.
KUGELGEN, Franz von (May 7, '04) Chief Chemist, The Va. Electrolytic Co., Holcomb's Rock, Va.
KUNITOMO, S. (July 30, '09) Chief Engineer, Joetsu Electric Co., Takata, Echigo, Japan.
KUNZ, Geo. F., Ph.D. (Sept. 28, '07) Gem Expert, Tiffany & Co., 401 Fifth Ave., New York City.
KWONG, KWANG YUNG (Apr. 24, '09) Directeur des Mines de Lincheng, Lin-tcheng-Sien, Pekin-Hankow Ry., Pet-Chili, North China (via Peking).
KYLE, T. D. (Apr. 3, '02) 106 E. 5th St., Leadville, Col.
LACROIX, Henry (Mar. 3, '06) Eng., Usine. de Degrossissage d'or, Geneve, Switzerland.
LAFORE, J. A. (Apr. 3, '02) 121 S. 11th St., Philadelphia, Pa.; res., Bala, Pa.
LAMB, Arthur B. (Dec. 27, '07) Associate Prof. of Chem., New York Univ., Univ. Heights, New York City.
LANDIS, Walter S. (Dec. 4, '02) Met.E., M.S., Asst. Prof. of Metallurgy, Lehigh Univ., res., 146 S. Linden St., Bethlehem, Pa.
LANDOLT, Dr. Hans (Feb. 5, '03) Turgi, Switzerland.
LANE, Henry M. (May 29, '09) Editor, "Castings," Caxton Bldg., Cleveland, Ohio.
LANGLEY, John W., Ph.D. (Apr. 3, '02) 2037 Geddes Ave., Ann Arbor, Mich.
LANGMUIR, Irving (June 29, '07) Research Lab., Genl. Elec. Co., Schenectady, N. Y.
LANGTON, John (Apr. 3, '02) Consulting Eng., 31 Nassau St. New York.
LANSLEY, William J. (Sept. 26, '08) Pres. and Consult. Eng., Hudson & Middlesex Telephone and Telegraph Co., Perth Amboy, N. J.
LARCHAR, Arthur B. (Apr. 3, '02) Penobscot Chem. Fibre Co., Great Works, Me.
LASS, W. P. (Mar. 27, '09) Alaska Treadwell Gold Mining Co., Treadwell, Alaska.
LATHROP, L. H. (Sept. 26, '08) Asst. Inspect. of El. Service, Wis. R. R. Comm., State Cap. Bldg., Madison, Wis.; mailing address, 602 E. Gorham St.
LAUGHLIN, H. Hughart (May 29, '09) Elec. Engr., Jones & Laughlin Steel Co., 2705 Carson St., Pittsburgh, Pa.; res., 5023 Bayard St.
LAVINO, Edward J. (Nov. 26, '07) E. J. Lavino & Co., Importers of Ferro-Alloys, Bullitt Bldg., Philadelphia, Pa.
Le BLANC, Prof. Dr. Max (Mar. 4, '05) Physikalisch-chemisches-Institut der Universitat, Leipzig, Germany.
LE BOUTILLIER, Clement (July 31, '08) Chem. and Met., Taylor Iron & Steel Co., High Bridge, N. J.
LEE, F. V. T. (Apr. 3, '02) 59th and College Ave., Oakland, Cal., assistant to President Pacific Gas and Elec. Co., San Francisco, Cal.
LEE, Harry R. (Dec. 2, '05) Holcomb's Rock, Va.
LE MARE, Ernest B. (Sept. 4, '03) c|o Pilkington Bros., Ltd., St. Helens, Lancashire, England.
LEWIS, JOHN B. (Sept 27. '07) Student, Univ. of Texas, Box 39, Univ. Station, Austin, Texas.
LEWIS, Jonathan D. (May 29, '09) Chemist, Edgar Thompson Wks., Carnegie Steel Co.; mailing address, 18 Center St., Braddock, Pa.
LICHTHARDT, G. (Apr. 3, '02) 18th and M Sts., Box 510, Sacramento, Cal.
LIDBURY, F. Austin (Aug. 7, '03) Works Mgr., Oldbury Electrochem. Co.; res., 33 Sugar St., Echota, Niagara Falls, N. Y.

LIEBMANN, Dr. Louis (Aug. 31, '07) Westend Str. 84, Frankfort a. Main, Germany.
LIENAU, J. Henry (Feb. 2, '07) Tech. Supt. New York Refinery, The Nat. Sugar Ref. Co. of N. J., Long Island City, N. Y.; res., 48 W. 82d St., New York City.
LIHME, C. B. (Nov. 27, '09) Chief Chemist, Illinois Zinc Co., La Salle, Ill.
LINCOLN, Dr. A. T. (Nov. 6, '02) Rensselaer Poly Inst.; res., 26 Second St., Troy, N. Y.
LINCOLN, Edwin S. (Sept. 26, '08), Consult. Eng., El. Goods Mfg. Co.; res., 132 Summitt Ave., Brookline, Mass.
LINCOLN, P. M. (Apr. 3, '02) 6830 Thomas St., Pittsburgh, Pa.
LIND, Samuel C. (July 30, '09) Instructor in General and Physical Chemistry, Chem. Lab., University of Michigan, Ann Arbor, Mich.
LINDSAY, Chas. F. (June 1, '07) Gen. Elec. Co., Schenectady, N. Y.
LITTLE, Arthur D. (Apr. 1, '05) Chem. Expert and Engineer, 93 Broad St., Boston, Mass.
LITTLE, C. A. (Apr. 3, '02) Electrochemist, 147 W. Bridge St., Elyria, Ohio.
LJUNGH, HJALMAR (Mar. 27, '09) Chemical Engineer, Copper Works, Falun, Sweden.
LLOYD, M. G., Ph.D. (Apr. 3, '02) Bureau of Standards, Washington, D. C.
LLOYD, Sherman C. (Sept. 26, '08) care of Deutsche Bank, Berlin, Germany.
LLOYD, Stewart A. (Oct. 28, '09) Adjunct Prof. of Metallurgy & Chem., University of Alabama, University, Ala.
LODYGUINE, A (Jan. 3, '04) Tverskaia 4, St. Petersburg, Russia.
LOEB, Morris (Apr. 3, '02) New York Univ., 273 Madison Ave., New York.
LOHMAN, R. W. (Apr. 6, '07) Eng. with Lohman & Co., Metropolis Bank. Bldg., San Francisco, Cal.
LONG, George E. (Jan. 28, '08) Asst. Chemist, Aluminum Co. of America, East St. Louis, Ill.
LOVE, Edward G. (May 29, '09) Consulting Chemist, Chief Gas Examiner, New York City; res., 80 E. 55th St.
LOVEJOY, D. R. (Apr. 3, '02) U. S. E. M. Co., 209 W. 33d St., New York City.
LUCKE, Henry J. (June 6, '03) c|o Edwards, Sager & Wooster, 2 Rector St., New York City.
LUNN, Ernest (Jan. 29, '09) Supt. of Storage Batteries, Comm. Ed. Co., 139 Adams St., Chicago, Ill.
LUNT, RAYMOND L. (Aug. 27, '09) Engineering Dept., Electric Storage Battery Co., 123 N. Central Ave., Chicago, Ill.
LYMAN, Jas. (Apr. 3, '02) Asst. Eng. Gen. Electric Co., 1047 Monadnock Bldg., Chicago, Ill.
LYON, Dorsey (Feb. 27, '09) Mgr. Noble Electric Steel Co., Heroult, Shasta County, Cal.
LYTLE, Clinton W. (June 25, '09) Mgr. and Supt., American Steel Foundries, Sharon, Pa.
MAC DONALD, Jas. A. (Aug. 7, '03) Vice-Pres. United Verde Copper Co., 49 Wall St., New York.
MAC GREGOR, Frank S. (Sept. 28, '07) Electrometallurgist, Huff Electrostatic Separator Co.; res., 60 India St., Boston, Mass.
MAC MAHON, Jas. (Aug. 7, '02) Bleaching Powder Supt., Castner Electrolytic Alkali Co., Niagara Falls, N. Y.
MAC NUTT, Barry, E.E., M.S. (Apr. 3, '02) Phys. Lab. Lehigh Univ.; res., 841 Seneca St., South Bethlehem, Pa.
MAGNUS, Benj. (Apr. 3, '02) Mgr. Electrolytic Refining and Smelting Co. of Australia, Port Kemble, N. S. W. Australia.
MAILLOUX, C. O. (Apr. 3, '02) 76 Williams St., New York.
MAIN, Wm. (Apr. 3, '02) Piermont, Rockland Co., N. Y.
MALM, John L. (Aug. 5, '05) El. Met., 1748 Welton St., Denver, Col.
MANAHAN, Paul R. (Apr. 30, '08) Mgr. Peabody Branch, Avery Chem. Co., 837 Caller St., Peabody, Mass.
MANTIUS, Otto (Jan. 28, '08) 615 Fidelity Bldg., Philadelphia.
MARIE, Charles A., Dr. es Sciences (Jan. 8, '04) rue de Cherche-Midi, Paris, VI, France.
MARSH, A. L. (May 29, '09) Chief Engineer, Hoskins Mfg. Co., 79 Rosedale Court, Detroit, Mich.
MARSH, Clarence W. (Dec. 26, '08) Chief. Eng., The Development & Funding Co., Niagara Falls, N. Y.
MARSH, Geo. E. (Oct. 28, '09) Instructor in Electrical Engineering, Armour Inst. of Tech., Chicago, Ill.
MARSHALL, Jas. G. (Sept. 2, '04) Asst. Supt. Union Carbide Co.; res., 1115 Niagara St., Niagara Falls, N. Y.
MARTI, William C. (Feb. 28, '08) Care of J. R. Francis, Chemist, Big Four Ry., Indianapolis, Ind.
MARTIN, Simon S. (Apr. 24, '09) Supt., Maryland Steel Co., Box 87, Sparrows Point, Md.
MARVIN, Arba B., Jr. (Apr. 3, '02) Patents and Patent Causes, 950 Commercial Natl. Bank Bldg., Chicago, Ill.
MASON, Edward J. K. (Mar. 27, '09) Prof. of Elec. Eng. and Physics, University of Pittsburgh, N. S., Pittsburgh, Pa.

MASTICK, Seabury C. (Feb. 27, '09) Binney, Mastick & Ogden, 2 Rector St., New York City.
MASUJIMA, Bunjiro (Mar. 4, '05) care of K. Takebe, 25 Gazenbo-cho, Azabuku, Tokyo, Japan.
MATHERS, Frank C. (Feb. 6, '04) 303 N. College Ave., Bloomington, Ind.
MATHEWS, Dr. John A. (May 29, '09) Manager, Halcomb Steel Co., Syracuse N. Y.
MATTHEW, H. T. (June 2, '06) 509 Old Colony Bldg., Chicago, Ill.
MAUELEN, Frederick (June 25, '09) Chemist, S. S. White Dental Mfg. Co., Prince Bay, Richmond Co., N. Y.
MAURAN, Max (Nov. 6, '02) Eng. and Asst. Mgr., Castner Electrolytic Alkali Co., Niagara Falls, N. Y.
MAYER, Frederick J. (Dec. 26. '08) Genl. Mgr., Didier-Mach. Co., 50 Church St. New York City.
MAYNARD, Geo. W. (Apr. 3, '02) 20 Nassau St., New York.
MAYS, E. A. (Nov. 27, '09) Chemist and Factory Superintendent, Robert A. Keasbey, New York City; mailing address, 442 W. 46th St.
MAYWALD, F. J. (Apr. 3, '02) Chem., 1028 72d St., Brooklyn, N. Y.
McALLISTER, Dr. Addams S. (July 31, '08) Assoc. Ed., The Electrical World, 239 W. 39th St., New York City.
McBERTY, Frank R. (Sept. 25, '09) Engineer, Western Electric Co., 463 West St., New York City.
McCLENAHAN, Howard (Apr. 24, '09) Prof. of Physics and Electrical Engineering, Princeton Univ., Princeton, N. J.; mailing address, 16 Stockton St.
McCONNELL, J. Y. (Apr. 3, '02) 500 N. Broad St., Philadelphia, Pa.; res., Colwyn, Pa.
McCORMACK, Harry (June 29, '07) Associate Prof. Dept. of Chemical Engineering, Armour Inst., Chicago, Ill.
McCOY, Herbert N. (Sept. 4, '03) Kent Chem. Lab., Univ. of Chicago, Chicago, Ill.
McELROY, Jas. F. (Sept. 17, '03) Consult. Eng., Cons. Car Heating Co., 131 Lake Ave., Albany, N. Y.
McFARLIN, J. Robert (Jan. 8, '08) Electrical Service Supplies Co., 1024 Filbert St., Philadelphia.
McINTOSH, D. (Dec. 4, '02) Demons. in Phys. Chem., McGill Univ., Montreal, Canada.
McKEE, Geo. M. (Aug. 7, '03) Mgr. Hinckley Fibre Co., Hinckley, Oneida Co., N. Y.
McKELVY, Ernest C. (Aug. 27, '09) Chemist, Bureau of Standards, Washington, D. C.
McKENNA, Chas. F. Ph.D. (Nov. 26, '07) 155 West 91st St., New York City.
McMURTRIE, Dr. Wm. (Apr. 6, '07) Royal Baking Powder Co.; res., 480 Park Ave., New York.
McNEILL, Ralph (Feb. 5, '03) 223 W. 106th St., New York.
MEDOVE, Morris (June 29, '07) c|o Wiederschall, 425 Grand St., New York City.
MELCHER, A. C. (July 3, '02) Research Associate in Phys. Chem., Mass. Inst. of Tech., Boston; res., 58 Bowen St., Newton Centre, Mass.
MEREDITH, William F. (May 29, '09) Titanium Alloy Mfg. Co., Niagara Falls, N. Y.
MERRILL, Prof. J. F. (Oct. 10, '03) Prof. of Physics and Elec. Eng., Dir. of State School of Mines, the Eng. School of Univ. of Utah, University of Utah, Salt Lake City, Utah.
MERRILL, J. L. (Sept. 4, '03) Local Agent, Burke Electric Co., 1526-30 Park Bldg., Pittsburgh, Pa.
MERSHON, Ralph D. (July 1, '05) Consult. Eng.; res., 116 W. 85th St., New York.
MERTON, Richard (Feb. 27, '09) Director, The Metallurgical Co. of America, 52 Broadway, New York City.
MERZ, Chas. H. (Apr. 3, '02) Consult. Eng., 28 Victoria St., Westminster, London, S.W.; Collingswood Bldgs., Newcastle-upon-Tyne, England.
MERZBACHER, Aaron (June 25, '09) Chemist, Crucible Steel Co. of America, Crescent Works, Pittsburgh, Pa.; mailing address, 217 Mathilda St.
METZ, H. A. (Apr. 3, '02) 122 Hudson St., New York.
MEYER, Dr. Franz (Sept. 4, '03) R. Wedekind & Co., m b H, Werdigen, Niederrhein, Germany.
MEYER, John (Oct. 7, '05) Eng. of Commercial Dept., Philadelphia, Elec. Co., Philadelphia, Pa.; res., 6746 Woodland Ave.
MILLENER, F. H. (Nov. 27, '09) Experimental Electrician, Union Pacific R. R., Omaha, Neb.; mailing address, Union Pacific Shops.
MILLER, Dr. W. Lash (Apr. 3, '02) 50 St. Albans St., Toronto, Canada.
MILLER, Levi B. (Jan. 29, '09) El. Chemist, G. E. Co., Harrison, N. J., res., 168 N. Grove St., East Orange, N. J.
MILLER, Walter H. (Mar. 27, '09) Mgr., Recording Dept., Edison Phonograph Co., Orange, N. J.; mailing address, 26 Linden Place.
MILLS, Harry P. (May 29, '09) Continental Mexican Rubber Co., Torreon, Coahuila, Mexico.
MILLS, James W. (Mar. 27, '09) Chem. and Metallurgist, Natl. Enameling & Stamping Co., Granite City, Ill., P. O. Box 15.
MILLS, J. E. (Apr. 16, '03) Chapel Hill, N. C.

MINER, H. S. (May 1, '07) Chief Chemist, Welsbach Light Co., Gloucester City, N. J.
MITCHELL, Thomas A. (Apr. 30, '08) West Va. Paper & Pulp Co., Mechanicville, N. Y.
MOLDENKE, Richard (Jan. 29, '09) Secty. and Treas., Am. Foundrymen's Assoc., Watchung, N. J.
MOLERA, E. J. (Apr. 3, '02) Civil Eng., 2025 Sacramento St., San Francisco, Cal.
MOLTKEHANSEN, Ivar J. (June 6. '03) 2 Avenue des Germains, Brussels, Belgium.
MONELL, A. (Apr. 3, '02) Pres. Int. Nickel Co., 43 Exchange Place, New York.
MONRATH, G. (Nov. 27, '09) Treasurer, American Development & Securities Co., New York City; mailing address, Singer Bldg.; res., 596 Riverside Drive.
MONTAGUE, Charles D. (Mar. 29, '09) Fredericksburg, Va.
MOODY, Herbert R. (June 29, '07) Chem. Bldg., College City of New York, 140th St. and Corwent Ave.; res., 23 Hamilton Terrace, College Station, New York City.
MORANI, Fausto (Dec. 4, '03) Gen. Mgr. Societa Italiana Carburo di Calcio Acetelene et Altri Gas, 66 Via Due Macelli, Rome, Italy.
MORDEN, Dr. G. W. (Feb. 27, '09) Manitoba Agricultural College, Manitoba, Winnipeg, Canada.
MOREHEAD, J. M. (Feb. 5, '03) 157 Michigan Ave., Chicago, Ill.
MOREY, George, Jr. (Nov. 26, '07) Care of Bureau of Standards, Washington, D. C.
MORGAN, Dr. J. L. R. (Apr. 3, '02) Columbia University, New York.
MORGAN, Leonard C. (Aug. 29, '08) Asst. Melter and Refiner, U. S. Mint, Philadelphia, Pa.
MORGANS, Frank Davis (Sept. 26, '08) Consult. Eng., 309 St. Louis St., El Paso, Texas.
MORITZ, C. H. (Apr. 4, '03) Aluminum Co. of America, Niagara Falls, N. Y.
MORRIS, Henry G. (Apr. 3, '02) London, Liverpool and Globe Bldg., 333 Walnut St., Philadelphia, Pa.
MORROW, John T. (Feb. 27, '09) Consulting Engr., Mexican Tramways Co., 25 Broad St., New York City.
MORSE, Willard S. (Jan. 29, '09) Dir. and Mem. Ex. Comm. Am. Smelting and Ref. Co., 165 Broadway, New York City.
MOTT, W. R. (Mar. 5, '03) 511 University Ave., Madison, Wis.
MOULTON, Chas. W. (May 1, '06) Prof. of Chem., Vassar College, Poughkeepsie, N. Y.
MOYER, Grant C. (Jan. 29, '09) Lab. Asst., FitzGerald & Bennie Labs., Niagara Falls, N. Y.
MULLER, H. N. (May 29, '09) Electrical Engineer, The Allegheny County Light Co., Pittsburgh, Pa.; mailing address, Cornell Ave. and Vassar Lane, N. S.
MURPHY, E. J. (Oct. 2, '02) 235 W. 108th St., New York city. (Care of Miss E. E. Blair.)
MURRAY, Benjamin L. (Nov. 27, '09) Chemist, Merck & Co., Rahway, N. J.; mailing address, 148 Bryant St.
MURRAY, HENRY T. (Apr. 24, '09) Chemist, Ray Consolidated Copper Co., Kelvin, Ariz.
MUSCHENHEIM, Frederick A. (Nov. 21, '08) Vice-Pres., Hotel Astor, New York City; res., 218 W. 45th St.
MYERS, Ralph E., Ph.D. (Nov. 5, '04) Chem. Engr., Westinghouse El. Co., Bloomfield, N. J.
NAMBA, M. (Nov. 6, '03) Prof. of Elec. Eng., Kyoto Imp. Univ., Kyoto, Japan.
NASH, Arthur D. (Apr. 24, '09) Asst Mgr. and Works Chemist, The Tiffany Furnaces, Flushing, N. Y.; mailing address, 280 State St.
NEIL, James M. (Dec. 27, '07) Technical Chemist, 12 Woodlawn Ave., Toronto, Canada.
NEWMAN, Frederick J. (Sept. 26, '08) Works Mgr., Woods Motor Vehicle Co., 2515 Calumet Ave., Chicago, Ill.
NICHOLS, Wm. H., D.Sc., LL.D. (Mar. 3, '06) Pres. Nichols Copper Co., General Chem. Co., 25 Broad St., New York; res., 355 Clinton Ave., Brooklyn, N. Y.
NICHOLS, W. Standish (Apr. 3, '02) Interborough Rapid Transit Co., 100 Broadway, New York.
NICKERSON, William E. (May 29, '09) Consulting Mechanical Engineer, Gillette Safety Razor Co., Cambridge, Mass.; mailing address, 1722 Massachusetts Ave.
NODELL, Wm. (May 9, '03) Elec. Eng., 10-12 E. 108th St., New York City.
NORMAN, Geo. M. (Apr. 3, '02) 75 Aberdeen Place, Woodbury, N. J.
NORTH, H. B. (April 6, '07) Care of Case School of Applied Science, Cleveland, Ohio.
NORTHRUP, Edwin F. (Oct. 17, '07) The Leeds & Northrup Co.; res., 8 Asbury Terrace, Oak Lane, Pa.
NUNN, Dr. R. J. (Apr. 3, '02) 5 York St., E., Savannah, Ga.
NUTTER, Coleman Evan (Oct. 7, '05) Elec. Eng., A., T. & S. F. Ry., Topeka, Kan.
OBER, Julius E. (Apr. 24, '09) West Penn. Steel Co., Keystone, Bldg., Pittsburgh, Pa.

ODDIE, James (Sept. 25, '09) Trustee and Life Governor, School of Mines, Ballarat, Victoria, Australia.
OESTERLE, Wm. F., Jr. (Oct. 7, '05) 404 Jarvis St., Clarkesburg, W. Va.
OHOLM, Lars Wm., Ph.D. (Dec. 27, '07) Lecturer on Applied Phys., Univ. of Finland, Helsingfors, Finland.
OLAUSSON, K. O. Ernfrid (Oct. 28, '09) Electrometallurgical Engineer, Aktiebolaget Saxberget, Trollhattan, Sweden.
ORNELLAS, Thomas V.D' (Nov. 21, '08) El. Eng., Peruvian Govt., Lima, Peru; mailing address, Calle de Lima 15, Charrillos.
ORNSTEIN G. (May 29, '09) Electrochemist, Development & Funding Co., Niagara Falls, N. Y.
ORR, R. S. (May 29, '09) General Superintendent, The Allegheny County Light Co., Pittsburgh, Pa.; mailing address, 435 6th Ave.
OSBORNE, Loyall A. (Apr. 3, '02) Westinghouse E. and Mfg. Co., Pittsburgh, Pa.
OSHIMA, Yoshikiyo Dr. Ing. (July 30, '09) Asst. Professor, Dept. of Chemistry, College of Engineering, Imperial University, Tokyo, Japan.
OSTHEIMER, John W. (June 25, '09) Student at the Ecole Centrale, Paris, France; 3 Rue Rabelais.
PAGE, George S. (May 29, '09) Asst. Mgr., Park Works, Crucible Steel Co. of America, Pittsburgh, Pa.
PALMER, Wm. R. (May 29, '09) General Supt., American Tube & Stamping Co., Bridgeport, Conn.; mailing address, 827 Seavind Ave.
PARKER, Herschel C. (July 31, '07) Adj. Prof. Physics, Columbia Univ., New York City; res., 21 Fort Greene Place, Brooklyn, N. Y.
PARKER, John C. (Nov. 21, '08) Mech. and El. Eng., Rochester Ry. & Lt. Co.; res., 34 Clinton Ave., N., Rochester, N. Y.
PARKHURST, C. W. (Sept. 26, '08) Supt. El. Dept., Cambria Steel Co., Johnstown, Pa.
PARR, Samuel W. (Jan. 7, '05) Prof. of Applied Chem., Univ. of Ill., Urbana, Ill.
PARSONS, Charles L. (Oct. 29, '08) Prof. of Inorganic Chemistry, New Hampshire College, Durham, N. Y.
PARSONS, Louis A., Ph.D. (Apr. 3, '02) Prof. of Physics, Pennsylvania College, Gettysburg, Pa.
PATTEN, Harrison E., Ph.D. (Apr. 3, '06) Bureau of Soils, U. S. Dept. of Agriculture, Washington, D. C.
PATTERSON, Andrew H. (Feb. 27, '09) Prof. of Physics, University of North Carolina, Chapel Hill, N. C.
PATTERSON, Geo. W., S.B., Ph.D. (Nov. 6, '02) Prof. of Elec. Eng., Univ. of Mich.; res., 814 S. University Ave., Ann Arbor, Mich.
PATTISON, Frank A. (Apr. 3, '02) Consult. Eng., Fuller Bldg., 23d St. and Broadway, New York.
PAUL, Henry N., Jr. (Apr. 3, '02) 1815 Land Title Bldg., Philadelphia, Pa.
PAWECK, Dr. Heinrich (Dec. 4, '03) Favoritenstrasse 60, Vienna IV, Austria.
PEARCE, John H. (Mar. 27, '09) Engineer, Elkhorn Electrometallurgical Co., Elkhorn, Mont.
PEARSON, Dr. Fred. S. (Sept, 25, '09) Consulting Engineer, 25 Broad St., New York City.
PEIRCE, Albert E. (Sept. 26, '08) Eng. and Asst. Gen. Mgr. Chippewa Valley Light and Power Co., Eau Claire, Wis.
PENNIE, John C. (May 29, '09) Attorney at Law and Patent Solicitor, 5 Nassau St., New York City.
PENNOCK, John D., A.B. (Apr. 2, '04) Chief Chemist, Solvay Process Co., Syracuse, N. Y.
PERKIN, F. Mollwo, Ph.D. (Sept. 4, '02) Prof. of Chem. Borough Poly. Inst., 103 Borough Road, S.E.; res., The Firs, Hengrave Road, Forest Hill, London, S.E., England.
PERRY, C. M. (Sept. 4, '03) Supt. Saylis Bleacheries and Glenlyon Dye Works, Saylesville, R. I.; res., Greene, R. I.
PERRY, R. S. (Apr. 3, '02) 5104 Pulaski Ave., Germantown, Philadelphia, Pa.
PETERSSON, Dr. Albert, Ph.D. Jan. 6, '06) Consult. Eng., Odda, per Bergen, Norway.
PETINOT, Napoleon (Apr. 24, '09) Electrometallurgical Engr., Niagara Falls, N. Y.; res., 632 Riverside Ave.
PEYTON, W. C. (Apr. 3, '02) Martinez, Contra Costa Co., Cal.
PHILIPP, Herbert (Nov. 6, '03) Roessler & Hasslacher Chem. Co.; res., 84 High St., Perth Amboy, N. J.
PHILLIPS, ROSS (Nov. 6, '03) National Electrolytic Co., Niagara Falls, N. Y.
PICKERING, Oscar W. (Oct. 2, '02) Consulting Chemist, 20 Lenox St., Springfield, Mass.
PIERCE, Robert F. (Oct. 29, '08) 30-32 E. 20th St., New York City.
PIKLER, A. Henry (June 1, '07) Engineer in charge Transformer Dept. Crocker Wheeler Co., Ampere, N. J.; res., Montclair, N. J.
PINKERTON, Andrew (Apr. 3, '03) Box 427, Pittsburgh, Pa.
POND, G. G., Ph.D. (July 3, '02) Prof. of Chem., State College, Pa.
PORTER, John L. (Apr. 3, '02) Lab. of Sewerage and Water Board, 602 Carondelet St.; res., 1317 Henry Clay Ave., New Orleans, La.

POTTER, Henry Noel, Sc.D. (Apr. 3, '02) Hollywood, Cal.
PRATT, Fred. S. (Apr. 3, '02) Stone & Webster, 93 Federal St., Boston, Mass.
PRICE, Edgar F. (July 3, '02) Union Carbide Co., 79 Wall St., New York City.
PRINDLE, Edwin J. (Jan. 8, '04) Practicing Patent Lawyer, 220 Broadway, New York.
PRING, John N. (Nov. 3, '06) Research student, Victoria Univ., Manchester, England.
PROEBSTEL, Chester L. (Feb. 27, '09) care of Proebstel Assaying Lab. and Metallurgical Lab., Yreka, Cal.
PROSSER, H. A. (Dec. 2, '05) Metallurg. Dir., U. S. Smelting, Ref. & Min. Co., 42 Broadway, New York City.
PUMPELLY, Jas. K. (July 1, '05) 2304 Ashland Ave., Indianapolis, Ind.
PYNE, Francis R. (Dec. 2, '05) U. S. Metals Ref. Co., Chrome, N. J.; res., 324 Union Ave., Elizabeth, N. J.
QUEENY, John F. (June 1, '07) President Monsanto Chem. Co., 1800 S. 2d St., St. Louis, Mo.
QUENEAU, A. L. J. (May 1, '06) Wetherill Finished Castings Co., Erie Ave. and Richmond St., Philadelphia, Pa.
QUINAN, Kenneth B. (Jan. 8, '04) Chief Chemist, De Beer's Explosive Works, Dynamite Factory, Somerset West, Cape Colony.
RAMAGE, A. S. (May 6, '05) The Electro Steel Co., 1545 Niagara St., Buffalo, N. Y.
RANDALL, J. W. H. (Jan. 29, '09) Chemist, El. Met. Co., 1100 Ferry Ave, Niagara Falls, N. Y.
RANDALL, Merle (Nov. 27, '09). Research Lab. of Physical Chem., Mass. Inst. of Tech., Boston, Mass.
RANDOLPH, Edward (Feb. 27, '09) Secretary and Treasurer, Balbach Smelting & Refining Co., Bretton Hall, New York City.
RAU, Prof. Albert G. (Apr. 3, '02) Supt. Moravian Parochial School, Bethlehem, Pa.
RAWLINS, J. Armstrong (May 29, '09) Vice-President, American Electric Furnace, 45 Wall St., New York City.
RAYNER, George R. (Mar. 27, '09) Secretary, The Carborundum Co., Niagara Falls, N. Y.
REBER, Lieut. Col. Samuel (Apr. 3, '02) 39 Whitehall St. (Army Bldg.) New York City.
REDMAN, L. V., B.A. (May 29, '09) Fellow in Electrochemistry, Toronto, University, Toronto, Canada.
REICHEL, Walter (Feb. 27, '09) Dr. Engr., 14 Beethovenstrasse, Lankwitz, Berlin, Germany.
REED, R. C. (May 29, '09) Supt., Elec. Dept., Carnegie Steel Co., Duquesne, Pa.
REED, C. J. (Apr. 3, '02) 3313 N. 16th St., Philadelphia, Pa.
REESE, P. P. (May 29, '09) Metallurgist, Carnegie Steel Co., Munhall, Pa.
REEVE, Amos G. (Nov. 26, '07) Electroplating Dept. Oneida Community, Ltd., Niagara Falls, N. Y.; res., 930 Ferry Ave.
REID, John T. (Mar. 27, '09) Mining Engineer, Nevada Mining Co., Lovelock, Humbolt Co., Nev.
REILLY, E. J., Jr. (May 29, '09) Chemist, Development & Funding Co., Niagara Falls, N. Y.; mailing address, 458 5th St.
REISENEGGER, Dr. Herman (June 6, '03) Chemiker Farbwerke in Hochst a|M, Germany.
REIST, Henry G. (Sept. 26, '08) Desig. Eng., Gen. El. Co., Schenectady, N. Y.
RHODIN, B. E. F. (Apr. 3, '02) Chief Eng., Can. Electrochem. Co., Ltd., Sault Ste. Marie, Ont., Canada.
RICHARD, George A. (June 1, '07) Electrolytic Ref. Co. of Australia, Mt. Morgan, Queensland, Australia.
RICHARDS, Edgar (Feb. 27, '09) Newport, R. I.
RICHARDS, Jos. W., Ph.D. (Apr. 3, '02) Prof. of Metallurgy, Lehigh Univ., S. Bethlehem, Pa.; res., University Park.
RICHARDS, Dr. T. W. (June 3, '05) Prof. of Chem., Harvard Univ., Cambridge, Mass.
RICHARDSON, Henry K. (May 29, '09) Prof. of Electrochemical Engineering, Penn. State College, State College, Pa.
RICHMOND, J. Stanley (Sept. 26, '08) Con. Eng., 34 Victoria St., Toronto, Canada; res., 48 Carlton St.
RICKENBACHER. Albert D. (Sept. 26, '08) Electrician for the City Post Office, Washington, D. C.; mailing address, 633 Keefer Place, N. W.
RIKER, John J. (Mar. 5, '03) Treas. Oldbury Electrochem. Co., 46 Cedar St., New York.
RIVA-BERNI, Count A. de (Nov. 26, '07) Compt. Int. de Vente de Ferro-Silicum; res., 9 Avenue Niel, Paris, France.
ROBB. C. D. (Nov. 27, '09) Partner, C. W. Leavitt & Co., New York City; mailing address, 30 Church St.; res., 210 W. 109th St.
ROBB, Wm. L. (Oct. 28, '09) Prof. of Electrical Engineering, Renssalaer Polytechnic Institute, Troy, N. Y.; mailing address, P. O. Box 592.

ROBERTS, Isaiah L. (Oct. 2, '02) Dir. Roberts' Chem. Co., 42 Broadway, New York.
ROBINSON, Almon (Apr. 3, '02); res., Webster Road, Lewiston, Me.
RODGERS, Ashmead G. (Nov. 6, '02) Supt. The Carborundum Co., Niagara Falls, N. Y.
RODMAN, Hugh (Apr. 3, '02) Westinghouse Machine Co., E. Pittsburgh, Pa.
ROEBER, E. F., Ph.D. (Apr. 3, '02) Ed. Electrochem. and Met. Ind., 239 W. 39th St., New York.
ROESSLER, Dr. Franz (July 31, '07) Vice-Pres. and Supt. Roessler & Hasslacher Chem. Co., Perth Amboy, N. J.; res., 39 High St.
ROGERS, Allen (Jan. 29, 09) Instr. in Indust. Chem., Pratt Institute, Brooklyn, N. Y.
ROGERS, Chas. M. (May 29, '09) Eng. Dept., Swift & Co., Box 713, Buffalo, N. Y.
ROLLER, F. W. (Apr. 3, '02) Elec. Instruments (Machado & Roller), 203 Broadway, New York.
ROSEBRUGH, Prof. T. R. (Apr. 3, '02) School of Practical Science, Toronto, Ont., Canada.
ROSS, G. McM. (Apr. 3, '02) Yosemite Club, Stockton, Cal.
ROSS, William (May 29, '09) Works Mgr., Susquehanna Smelting Co., Lockport, N. Y.
ROSSI, A. J. (Apr. 3, '02) 35 Broadway, New York.
ROUSE, Edwin W., Jr. (Feb. 27, '09) Chemist, Baltimore Copper, Smelting and Rolling Co., Baltimore, Md.
ROUSH, Gar G. (Feb. 6, '04) Gas City, Ind.
ROWAND, Lewis G. (Apr. 3, '02) N. J. Zinc Co., Franklin Furnace, Sussex Co., N. J.
ROWLAND, Thomas (Feb. 27, '09) Mgr., American Electric Furnace Co., Box 347, Niagara Falls, Canada.
RUDRA, Sarat C. (Mar. 5, '04) Consult. Min. Eng., 2963 Webster St., San Francisco, Cal.; res., Calcutta, India.
RUHL, Louis (Dec. 2, '05) Asst. Sec. The Roessler & Hasslacher Chem. Co., 100 William St., P. O. Box 1999, New York.
RUPPEL, Henry E. K. (May 29, '09) Chemist, Gilette Razor Co., South Boston, Mass.
RUSHMORE, David B. (Apr. 3, '02) Eng. Power and Mining Dept., General Elec. Co., Schenectady, N. Y.
RUSSELL, Chas. J. (Apr. 3, '02) District Manager The Phila. Electric Co., 4522 Frankford Ave., Philadelphia, Pa.
RUTHENBURG, Marcus (Apr. 3, '02) 34 Clements Lane, London, England.
RYNARD, Wm. T. (Aug. 5, '05) Gen. Mgr. Vanadium-Alloys Co., 25 Broad St.; res., 116 Riverside Drive, New York.
SADTLER, Samuel P., LL.D. (Apr. 3, '02) Consulting Chemist, 39 S. 10th St., Philadelphia, Pa.
SADTLER, Samuel S. (Apr. 3, '02) with S. P. Sadtler & Son, Consulting and Analytical Chemists, 39 S. 10th St., Philadelphia, Pa.
SALOM, Pedro G. (Apr. 3, '02) 333 Walnut St., Philadelphia, Pa.
SANG, Alfred (Dec. 26, '08) 96 Boulevard de Versailles, Saint Cloud (Seine-et-Oise), France.
SARGENT, Frank C. (Jan 29, '09) El. Eng., Malden Electric Co., Malden, Mass.
SAUNDERS, Prof. A. P. (Apr. 3, '02) Hamilton College, Clinton, N. Y.
SAUNDERS, Lewis E. (Dec. 26, '07) Chem. Eng., Norton Co., Box 296, Niagara Falls, N. Y.
SAUNDERS, Walter M. (Mar. 27, '09) Saunders & Franklin, Providence, R. I.; mailing address, 20 Dewey St.
SAYERS, Royd R. (June 1, '07) Lancaster, N. Y.
SCHALL, Dr. C. (Aug. 29, '08) Privatdocent, Sternwarterstrasse 79|1, Leipzig, Germany.
SCHAMBERG, Meyer (Dec. 27, '07) Chemist and Min. Engr., 1841 N. 17th St., Philadelphia, Pa.
SCHILDHAUER, Edward (Dec. 26, '08) El. and Mech. Eng., Culebra, Canal Zone, Panama.
SCHILLER, F. W. (Sept. 26, '08) Insp. Pub. Service Comm., State of N. Y.; res., 168 Seymour Ave., Utica, N. Y.
SCHLESINGER, Barthold E. (Nov. 6, '02) The Merrimac Chem. Co., res., 92 Mt. Vernon St., Boston, Mass.
SCHLOSS, Joseph A. (Jan. 29, '09) Ore Buyer, 42 Broadway, New York City.
SCHLUEDERBERG, Carl G. (Feb. 2, '06) 210 N. Craig St., Pittsburgh, Pa.
SCHLUNDT, Herman (Nov. 5, '04) Asst. Prof. Phys. Chem., Univ. of Missouri; res., 801 College Ave., Columbia, Mo.
SCHOCH, Dr. Eugene P. (Oct. 1, '04) Adjunct Prof. of Chem., Univ. of Texas; res., 714 W. 23d St., Austin, Tex.
SCHROEDER, C. M. Edward (May 29, '09) Chief Assistant, Dr. C. F. McKenna, 235 Wood St., Rutherford, N. J.
SCHRODER, Harold (Jan. 29, '09) Metallurgist, El. Ref. & Smelt. Co., Port Kembla, N. S. W., Australia.
SCHUETZ, Fred. F. (Oct. 1, '04) Patent Attorney, Room 1352, 50 Church St., New York City.

SCOTT, Arthur P. (Mar. 27, '09) Metallurgist, Research Laboratory, General Electric Co., Schenectady, N. Y.
SCOTT, CHAS. F. (Aug. 27, '09) Consulting Engineer, Westinghouse Electric & Mfg. Co., Pittsburgh, Pa.
SCOTT, Walter H. (June 25, '09) Chemist and Electric Furnace Assistant, Halcomb Steel Co., Syracuse, N. Y.; res., 204 Summit Ave.
SCUDDER, Heyward (Sept. 4, '03) 21 E. 22d St., New York.
SEABURY, R. W. (Nov. 27, '09) Secretary and General Manager, Boonton Rubber Co., Boonton, N. J.
SEEDE, John A. (May 29, '09) Electrical Engineer, General Electric Co., Schenectady, N. Y., P. O. Box 1048.
SEIDEL, Carlos M. (Nov. 27, '09) Oficios No. 22, Havana, Cuba.
SERGEANT, Elliott M. (October 1, '04) Box 54, Niagara Falls, N. Y.
SETHMAN, Geo. H. (Nov. 6. '03) Engineer and Contractor, 125 E. 11th Ave., Denver, Colo.
SEWARD, Geo. O. (April 3, '02) 99 Cedar St., New York.
SEYFERT, Stanley S. (Oct. 29, '08) Asst. Prof. El. Eng., Lehigh Univ.; res., 530 Chestnut Street, South Bethlehem, Pa.
SHARP, Clayton H. (Nov. 26, '07) Test Officer with Electrical Testing Laboratories, 80th St. and East End Ave., New York City; res., White Plains, N. Y.
SHARPLESS, Stephen S. (Feb. 27, '09) Consulting Chemist, Campbell Magnetic Separator Co., Boston, Mass.; mailing address, 26 Broad St.
SHATTUCK, A. F. (Apr. 3, '02) The Solvay Process Co., Detroit, Mich.
SHAW, E. C. (Apr. 3, '02) The B. F. Goodrich Co., Akron, Ohio; res., 120 S. Union St.
SHAW, Prof. H. B. (Apr. 3, '02) Univ. of Missouri, Columbia, Mo., Prof. of Elec. Engineering.
SHELDON, Dr. Samuel (Apr. 3, '02) Prof. of Physics and Elec. Eng., Polytechnic Inst., Brooklyn, N. Y.
SHIELDS, Dr. John (Dec. 4, '02) 4 Stanley Gardens, Cricklewood, London, N. W., England, and Minas de Rio Tinto, Provincia de Huelva, Spain.
SHINJO, Y. (Nov. 5, '04) Chief Eng. Tokyo Elec. Co., 1 Amishirocho, Azabuku, Tokyo, Japan.
SHINN, F. L. (Jan. 8, '04) Asst. Prof. of Chem., Univ. of Oregon; res., 386 E. 11th St., Eugene, Ore.
SHIVERICK, Myron D. (Nov. 21, '08) Electrician and Chemist, H. E. Milbank & Co., Albany, N. Y.; res., 30 Lancaster St.
SIMMERS, Arthur L. (May 29, '09) Chief Draftsman, Dobby Foundry and Machine Co., P. O. Box 758, Niagara Falls, N. Y.
SIMPSON Louis (May 29, '09) Representative, Electro-Metal Co., of Ludvika, Fitzroy Harbor., Ont., Canada.
SJOSTEDT, E. A. (Sept. 4, '03) Chief Metallurgist, The Lake Superior Power Co., Sault Ste. Marie, Ont., Canada.
SKINNER, H. J. (Apr. 3, '02) 93 Broad St., Boston, Mass., c|o A. D. Little.
SKOWRONSKI, Stanislaus (June 1, '07) Care of C. L. Constant & Co., 42 New St., New York City.
SLADE, W. C. (Apr. 30, '08) Dept. of Chemistry, Brown Univ., Providence, R. I.
SLOCUM, Frank L. (Dec. 4, '03) Part Owner, Vice-Pres. and Gen. Mgr., Miami Min. Co., 401 S. Linden Ave., E. E., Pittsburgh, Pa.
SMITH, A. T. (Dec. 4, '03) Gen. Mgr. Castner-Kellner Alkali Co., 43 Castle St., Liverpool, England.
SMITH, Prof. A. W. (Apr. 3, '02) Case School of Applied Science; res., 1971 E. 79th St., N. E., Cleveland, Ohio.
SMITH, Clement A. (Dec. 26, '08) El. Eng., 3 Warwick Road, Sparkhill, Birmingham, Eng.; care of Mrs. Connell.
SMITH, E. A. C. (May 9, '03) Metallurgist, Balt. Cop Smelting and Rolling Co., Baltimore, Md.
SMITH, Dr. Edgar F. (June 3, '05) Prof. of Chem., Univ. of Pa., Philadelphia, Pa.
SMITH, Edmund S. (Apr. 3, '02) Chemist, The Carborundum Co., Niagara Falls, N. Y.
SMITH, E. W. (Apr. 3, '02) 74 E. Penn St., Germantown, Philadelphia, Pa.
SMITH, F. Warren (May 9, '03) Provo City, Utah.
SMITH, Prof. Harold B. (Sept. 4, '03) Prof. of Elec. Eng., Worcester Polytechnic Inst.; res., 20 Trowbridge Road, Worcester, Mass.
SMITH, Wm. (Nov. 4, '05) Mech. Eng., Henry Disston & Sons, Inc.; res., 6942 Marsden St., Tacony, Philadelphia, Pa.
SMITH, Wm. Acheson (Aug. 31, '07) Vice-Pres. Inter. Acheson Graphite Co.; res., 603 Jefferson Ave., Niagara Falls, N. Y.
SMULL, Judson G. (Nov. 26, '07) 120 W. Broad St., Bethlehem, Pa.
SMYTHE, Clarence V. (Mar. 27, '09) Elect. Engr., care of Messrs. R. Ditmar, 55 Radha Bazar, Calcutta, Ind; res., 5 Ripon St. (West).
SMYTHE, Edwin H. (May 29, '09) Electrical Engineer, 738 Monadnock Bldg., Chicago, Ill.
SNELLING, W. O. (Oct. 2, '02) U. S. Testing Station, 40th and Butler Sts., Pittsburgh, Pa.
SNOOK, H. Clyde, A.M. (Nov. 26, '07) Pres. Roentgen Mfg. Co., Mariner and Merchant Bldg., Philadelphia, Pa.

SNOWDON, R. C. (Feb. 2, '06) 77 Water St., Ossining, N. Y.
SNYDER, Fred. T. (June 6, '03) Pres. Canada Zinc Co., 738 Monadnock Bldg.,
SOLVAY, Armand (Jan. 8, '04) Mgr. The Solvay Co., of Brussels, 33 rue du Prince Albert, Brussels, Belgium.
SONNHALTER, Aaron L. (May 29, '09) Chemist, American Steel Foundries, Sharon, Pa.
SPALDING, William L. (Apr. 30, '08) Supt. Electrolytic Ref., Buffalo Smelt. Wks., Buffalo, N. Y.
SPEIDEN, Clement C. (Nov. 6, '02) 46 Cliff St., New York.
SPEIDEN, Eben C. (Feb. 2, '06) Supt. Int. Acheson Graphite Co., Niagara Falls, N. Y.
SPERRY, Elmer A. (Apr. 3, '02) 100 Marlborough Road, Prospect Park, South, Brooklyn, N. Y.
SPERRY, Erwin S. (Aug. 27, '09) Editor, The Brass World and Platers' Guide, Bridgeport, Conn.; mailing address, 260 John St.
SPICE, Robert (June 6, '03) Prof. of Chem., Cooper Union for the Advancement of Science and Art, New York.
SPRAGUE, Edmund C. (July 31, '08) Chemist, Int. Acheson Graphite Co.; res., 518 Jefferson Ave., Niagara Falls, N. Y.
SPURGE, Edward C. (May 29, '09) Manager, Ozone Vanilin Co., Niagara Falls, N. Y.; mailing address, University Club.
STAMPS, F. A. (June 2, '06) Chem. Phosphorus Compounds Co., P. O. Box 250; res., 521 Jefferson Ave., Niagara Falls, N. Y.
STANLEY, Wm. (Jan. 8, '04) Great Barrington, Mass.
STANSFIELD, A., D.Sc., A.R.S.M. (Jan. 8, '03) Prof. of Metallurgy, McGill Univ., Montreal, Canada.
STANTON, McM. F. (Nov. 27, '09) Agent, Atlantic Mining Co.; res., Atlantic Mine, Mich.; mailing address, 208 5th Ave., New York City.
STARK, Edgar E. (Jan. 29, '09) City Electrician, Dunedin, New Zealand; (Box 465).
STASSANO, Maj. Ernesto (Sep. 25, '09) Managing Director, Forni Elettrici Stassano, Turin, Italy.
STATHAM, Noel (Oct. 17, '07) W. Va. Pulp and Paper Co., Mechanicville, N. Y.; res., 164 S. Main St.
STEIN, Walter M. (Jan. 8, '03) Pres. and Gen. Mgr. Primos Chem. Co., Primos, Delaware Co., Pa.
STEINMETZ, Charles P. (Aug. 7, '02) Gen. Elec. Co.; res., Wendell Ave., Schenectady, N. Y.
STEINMETZ, J. A. (Apr. 3, '02) 401 Market St., Philadelphia, Pa.
STEVENS, R. H. (May 29, '09) Mechanical Engineer, Carnegie Steel Co., Homestead Works, Munhall, Pa.
STEVENS, J. Franklin (Sept. 26, '08) Pres. Keystone El. Inst. Co., Ninth street and Montgomery Ave., Philadelphia, Pa.
STEVENS, Oscar E. (Sept. 26, '08) Gen. Ry. Signal Co., 45 Broadway, New York City.
STEWART, Robert Stuart (Sept. 26, '08) Consult. El. Eng., 814 Penobscot Bldg., Detroit, Mich.
STITCHT, Robert C. (May 29, '09) General Mgr., Mt. Lyell M. & R. Co., Ltd., Queenstown, Tasmania, Australia.
STILLMAN, Dr. T. B. (Sept. 4, '03) Prof. of Engineering Chem., Stevens Inst. of Tech., Hoboken, N. J.
STINCHFIELD, Ray L. (Dec. 26, '08) Asst. Exam., U. S. Pat. Office, Div. 14, Washington, D. C.
STOLL, Clarence G. (Mar. 27, '09) Engineer of Methods, Western Electric Co., 463 West St., New York City.
STONE, Chas. A. (Apr. 3, '02) Stone & Webster, 84 State St., Boston, Mass.
STONE, Edgar D. (Feb. 27, '09) General Mgr., Georgia-Tennessee Phosphate Co., Boma, Putnam Co., Tenn.
STONE, George W. (Mar. 27, '09) Assistant Superintendent, The Development & Funding Co., Niagara Falls, N. Y.; mailing address, 14 C St., Echota, N. Y.
STONE, I. F. (Jan. 29, '09) Pres., Nat. Analine & Chem. Co., 100 William St., New York City.
STONE, Joseph P. (Nov. 21, '08) El. Eng., Agar Cross y Cia, 949 Calli Cuijo, Buenos Aires, Argentine Republic, S. A.
STONE, John Stone (Jan. 29, '09) Pres., Stone Tel. & Tel. Co., Boston, Mass.; res., 192 Bay State Road.
STOVEL, R. W. (Sept. 26, '08) Eng., West., Church, Kerr & Co., 10 Bridge St., New York City.
STOVER, Edward C. (Dec. 26, '08) Asst. to Gen. Mgr., Trenton Potteries Co., Trenton, N. J.: res., 474 W. State St.
STRONG, Archie H. (May 29, '09) Correspondent, The American Electric Furnace Co., 45 Wall St., New York City.
STROSACKER, C. J. (Sept. 26, '08) Foreman of Sodium Benzoate Plant, Dow Chem. Co., Midland, Mich.
STURGESS, F. M. (Sept. 26, '08) Apprentice, West. El. & Mfg. Co.; res., 274 W. Swissvale Ave., Swissvale, Pa.

STUTZ, Ernest (Apr. 7, '04) Vice-Pres. and Gen. Mgr. Goldschmidt Thermit Co., 90 West St., New York.
SUMMERS, Leland L. (Sept. 26, '08) L. L. Summers & Co., First Nat'l Bank Bldg., Chicago, Ill.
SYKES, H. Walter (May 7, '04) 503 Midland Ave., Syracuse, N. Y.
SYMMES, Whitman (Dec. 4, '03) Consult. Chem. Eng., Ward Shaft Association, Virginia City, Nev.
TADA, Lieut. S. (Sept. 4, '03) Kure Naval Arsenal, Kure, Japan.
TAGGART, Walter T. (Nov. 6, '02) Asst. Prof. in Organic Chem., Univ. of Pa.; res., 717 Corinthian Ave., Philadelphia, Pa.
TAKAHASHI, Taketaro (July 30, '09) Yoshida, Kamimizuchigun, Naganoken, Japan.
TAKAMINE, Jokichi (Feb. 27, '09) Research Chemist, Parke Davis & Co., 550 W. 173d St., New York City.
TALBOT, Prof. H. P. (Jan. 8, '04) Prof. of Inorganic and Analyt. Chem., Mass. Inst. of Tech.; res., 273 Otis St., West Newton, Mass.
TATE, John V. E. (Mar. 27, '09) Chief Line Inspector, The Cauvery Elec. Power Scheme, Kankanhalli, Mysore State, India.
TAYLOR, Charles E. (May 29, '09) Chemist for Edward R. Taylor, Penn Yan, N. Y.; (Drawer M).
TAYLOR, Edward R. (Apr. 3, '02) Penn Yan, N. Y.
TAYLOR, W. T. (Dec. 26, '08) "Glendower," 76 Greenham Road, Muswell Hill, London, England.
TEEPLE, Dr. J. E. (May 1, '06) Consult. and Mfg. Chem., 50 Church St., New York.
THATCHER, C. J., Ph.D. (Jan. 7, '05) Pat. Exp., 50 Church St., New York City.
THOMAS, Benj. F. (June 6, '03) Sec. Coco Cola Bottling Co., The Administration and Trust Co. Bldg., Chattanooga, Tenn.
THOMAS, Royal D. (Mar. 2, '07) Asst. to Elec. Eng. Moorehead Bros., Sharpsburg, Pa.; res., Oakmont, Pa.
THOMPSON, Prof. Elihu (Sept. 17, '03) Elec., Gen. Elec. Co., 22 Monument Ave., Swampscott, Mass.
THOMPSON, JOHN (Feb. 27, '09) Civil and Mechanical Engineer, 253 Broadway, New York City.
THOMPSON, M. deK., Jr. (Nov. 6, '03) Instr. in Electrochem., Mass. Inst. of Tech., Boston, Mass.
THOMPSON, Robert M. (Apr. 3, '02) 41-43 Wall St. and 43-49 Exchange Place, New York.
THRELFALL, Richard (Apr. 4, '03) 30 George Road, Birmingham, England.
THWING, C. B. (Nov. 27, '09) 5 Cresheim Road, Philadelphia.
TIDD, George N (June 23, '08) Care of American Gas & Electric Co., 30 Church St., New York City.
TILLBERG, Erik W. (Nov. 6, '02) Westervik, Sweden.
TOCH, Maximilian (Nov. 6, '03) 320 Fifth Ave., New York.
TOMLINSON, L. C. (Oct. 29, '08) Care Automatic Elec Co., Morgan and Van Buren Sts. Chicago, Ill.
TONE, F. J. (Apr. 3, '02) Works Mgr., The Carborundum Co., Niagara Falls, N. Y.
TOPPING, Alanson N. (Sept. 26, '08) Asst. Prof. El. Eng., Purdue University, Lafayette, Ind.; res., 1022 1st St., West Lafayette.
TOWNSEND, C. P. (Apr. 3, '02) 918 F St., N. W., Washington, D. C.
TUCKER, Samuel A. (Mar. 3, '06) Adjunct Prof. Electrochem., Columbia Univ.; res., 155 E. 61st St., New York.
TUFTS, JOHN L. (June 1, '07) Chem. Eng., Merrimac Chem Co., North Woburn, Mass.; mailing address, 15 Park Ave., Winchester, Mass.
TURNBULL, Robert (Feb. 27, '09) Engr. with P. L. T. Heroult, 23 Duke St., St. Catherines, Ont., Canada.
TURRENTINE, John W. (Oct. 29, '08) Inst. in Phys. and Electrochem., Wesleyan Univ., Middletown, Conn.
ULKE, Titus (Apr. 3, '02) U. S. Patent Office, Washington, D. C.
UNGER, M. (Nov. 27, '09) Electrical Engineer, The General Electric Co., Pittsfield, Mass.; mailing address, 37 Springside Ave.
VALENTINE, Wm. (Sept. 17, '03) 22 Mitchell Ave., Waterbury, Conn.
VAN WINCKEL, W. H. (Dec. 1, '06) The Dow Chemical Co., Midland, Mich.
VAN WINKLE, Abr. (Jan. 8, '03) Pres. The Hanson & Van Winkle Co., 35 Lincoln Park, Newark, N. J.
VAUGHN, C. F. (Nov. 6, '02) Supt. Castner Electrolytic Alkali Co., University Club, Niagara Falls, N. Y.
VIAL, F. K. (May 29, '09) Consulting Engineer, Griffin Wheel Co., Sacramento, Square, Chicago, Ill.
VICTORIN, Anthony (Mar. 27, '09) Consulting Engr., Hotel Imperial, Broadway and 31st St., New York City.
VILLARELLO, Juan D. (Mar. 27, '09) Assistant Director, Instituto Geologico de Mexico, 3th Pino 129, Mexico City, Mexico.
VOEGE, Adolph L. (Apr. 3, '02) Concilium Bibliographicum, Hofstrasse, Zurich, Switzerland.
VOGEL, G. C. (Apr. 3, '02) Pfister & Vogel, Milwaukee, Wis.
VOORHEES, Louis A. (Apr. 3, '02) 111 Carroll Place, New Brunswick, N. J.

VORCE, L. D. (July 31, '07) Supt. Penna. Salt Mfg. Co., Wyandotte, Mich.
VROOMAN, H. H. (June 25, '09) Engr. Power Testing Dept., Mexican Light & Power Co., 3a Tobasco No. 75, Mexico City, Mexico.
WADDELL, Montgomery (Mar. 27, '09) Consult. Engr., 1 W. 101st St., New York City.
WADSWORTH, Frank L. O. (July 1, '04) Sewickley, Pa.
WAGNER, Henry L. (Apr. 3, '02) Prof. of Laryngology in San Francisco Polyclinic, 2339 Bush St., San Francisco, Cal.
WAGNER, Prof. Dr. J. (Dec. 4, '02) Leipzig, Mozartstrasse 7, Germany.
WALDMAN, Louis I. (Oct. 17, '07) Pres. Hudson River Aniline Color Works, P. O. Box 688, Albany, N. Y.
WALDO, Leonard (Nov. 4, '05) Consult. Eng., 49 Wall St., New York.
WALES, S. S. (May 29, '09) Supt. Armor Plate Dept., Carnegie Steel Co., Munhall, Pa. (P. O. Box 723).
WALKER, Arthur L. (Feb. 27, '09) Prof. of Metallurgy, Columbia University, New York City.
WALKER, C. R. (Mar. 5, '03) 266 Grand St., Newburgh, N. Y.
WALKER, Dr. J. W. (Dec. 4, '02) Prof. of Chem., Chem. and Min. Bldg., McGill Univ., Montreal, Canada.
WALKER, Dr. W. H. (Aug. 7, '02) Prof. Ind. Chem., Mass. Inst. of Tech., Boston, Mass.
WALLACE, Walter (Jan. 29, '09) Chemist, Oldbury El. Chem Co., Niagara Falls, N. Y.
WALSH, Philip C., Jr. (Apr. 7, '06) Member Walsh's Sons & Co., metal and machinery merchants, 19 Grant St., Newark, N. J.
WALTERS, Harvey E. (Sept. 28, '07) Electrician, Penna. R. R. Co., Altoona, Pa.; res., 410 16th St.
WALTON, J. M. (Nov. 27, '09) Storage Battery Engineer, Edison Elec. Ill. Co., Brooklyn, N. Y.; mailing address, 616 Carlton Ave.
WARD, Louis E. (March 25, '08) 524 State St., Madison, Wis.
WARING, Tracy D. (Apr. 6, '07) Standard Underground Cable Co., Perth Amboy, N. J.
WARREN, George S. (June 25, '09) Mechanical Engr., Sharon Steel Hoop Co., Sharon, Pa.
WASHBURN, Frank S. (Oct. 29, '08) Pres. Amer. Cyanamid Co., Stahlman Bldg., Nashville, Tenn.
WATERMAN, Frank N. (Apr. 3, '02) 100 Broadway, New York.
WATSON, Ralph A. (Feb. 27, '09) Electrical Engineer, Watson & Dey, Apartado No. 2731, Mexico City, Mexico.
WATTS, Geo. W. (Oct. 28, '09) Works Manager, The Canadian General Elec. Co., Ltd., Toronto, Canada; res., 1 Beaumont Road.
WATTS, Oliver P., Ph.D. (Mar. 5, '04) Asst. Prof. of Applied Electrochem., Univ. of Wisconsin; res., 313 Johnson Court, Madison, Wis.
WEAVER, W. D. (Apr. 3, '02) Editor, Electrical World, 239 W. 39th St., New York; res., 55 Demarest Ave., Englewood, N. J.
WEBB, L. W. (Sept. 26, '08) Master Elect., Const. and Rep. Dept., Navy Yard, Norfolk, Va.
WEBSTER, Clement L. (Apr. 24, '09) Geologist and Mining Engr., The Interstate Investment and Development Co., Charles City, Floyd Co., Iowa; mailing address, 111 Hawkins Ave.
WEBSTER, Edwin S. (Apr. 3, '02) Stone & Webster, 84 State St., Boston, Mass.
WEEDON, Dr. W. S. (April 3, '02) E. I. du Pont de Nemours Powder Co., Du Pont Bldg., Wilmington, Del.
WEEKS, Chas. A. (Nov. 27, '09) Mechanical Engineer, 810 Provident Bldg., Philadelphia; res., 2006 N. 20th St.
WEIMER, Edgar A. (Jan. 29, '09) Pres. and Supt., Weimer Machine Co., Lebanon, Pa.
WEINTRAUB, Ezechiel (Oct. 29, '08) Director of Research Lab., Gen. El. Co., West Lynn, Mass.
WELD, Harold K. (Feb. 27, '09) Sales Dept. McRoy Clay Works, Room 445, The Rookery, Chicago, Ill.; mailing address, 313 Maple Ave., Oak Park, Ill.
WELLENSIEK, A. H. (Sept. 26, '08) El. Eng., Easton & Wellensiek, Aberdeen, S. D.
WELLS, G. A. (Sept. 2, '05) Chief Eng., Adams Express Co., 61 Broadway, New York.
WELLS, Prof. J. S. C. (Apr. 3, '02) 320 W. 83d St., New York City.
WELLS, Roy (Feb. 27, '09) Electrician, Montana Power Transmission Co., Divide, Mont.
WERLIIN, Godwin (Sept. 25, '09) Electrical Engineer, Henry Bower Chem. Mfg. Co., 29th St. and Grays Ferry Road, Philadelphia; res., Moore, Delaware Co., Pa.
WESSON, David (May 1, '06) Mgr. Tech. Dept., The Southern Cotton Oil Co., 111 S. Mountain St., Montclair, N. J.
WESTON, Edward, Sc.D., LL.D. (Apr. 3, '02) 645 High St., Newark, N. J.
WETZLER, Jos. (Jan. 8, '03) Pres. The Electrical Eng. Inst., 240 W. 23d St.; res., 257 W. 104th St., New York.

WEYMOUTH, Frederick A. (May 29, '09) Special Metallurgist, Maryland Steel Co., Sparrows Point, Md. (P. O. Box 43).
WHEELER, Archer E. (Feb. 6, '04) Supt. of Reduction Works, Boston and Mont. Cons. Cop. and Silver Min. Co., Great Falls, Mont.
WHEELER, Fred. B. (Apr. 24, '09) Gas Engineering Expert, Semet-Solvay Co., Syracuse, N. Y.
WHITE, Edmond A. (July 31, '08) Const. Eng., The Electrolytic Ref. and Smelt. Co., Port Kembla, N. S. W. Australia.
WHITE, E. E. (Mar. 27, '09) Care of Cleveland-Cliffs Iron Co., Ishpeming, Mich.
WHITE, J. G. (Sept. 4, '03) Pres. J. G. White & Co., 43-49 Exchange Place, New York; Chairman of J. G. White Co., Ltd., 22A College Hill, London, E.C., England; res., 440 West End Ave., New York.
WHITE, Marcus (Feb. 27, '09) The Iva Grace M. Co., Silver City, Owyhee Co., Idaho.
WHITE, Ray Hill (Oct. 2, '02) 83 East Ave., Rochester, N. Y.
WHITING, Jasper (Nov. 26, '07) 15 Beason St., Boston, Mass.
WHITING, S. E. (Apr. 3, '02) Boston Elevated Railway Co.; res., 11 Ware St., Cambridge, Mass.
WHITLOCK, E. H. (May 9, '03) Asst. Supt. National Carbon Co.; res., Open Hearth, Lake Ave., Cleveland, Ohio.
WHITNEY, Dr. W. R. (Apr. 3, '02) General Elec. Co., Schenectady, New York.
WHITTEN, Wm. M. (Apr. 3, '02) 727 Du Pont Bldg., Wilmington, Del.
WICHMANN, Hugo J. (Dec. 27, 07) 2415 Avenue K, Galveston, Tex.
WIDMANN, E. A. (Nov. 27, '09) Secretary, H. A. Metz & Co., P. O. Box 58, New York City; res., 495 8th Ave., Brooklyn, N. Y.
WIECHMANN, F. G. (Apr. 3, '02) 24 State St., New York City.
WIGGLESWORTH, Henry (June 6, '03) Mfg. Chem., General Chem. Co., 25 Broad St., New York.
WILDER, F. L. (Feb. 6, '04) Chief Metallurgist, St. John d'el Rey Min. Co., Ltd., Morro Velho, Villa Nova de Lima, Minas Geraes, Brazil.
WILEY, Brent (May 29, '09) Commercial Engineer, Westinghouse Elec. & Mfg. Co., Pittsburgh, Pa.; mailing address, 652 Maryland Ave.
WILEY, Dr. H. W. (Jan. 8, '03) Chief of Bureau of Chem., Dept. of Agriculture, Washington, D. C.
WILKE, William (Feb. 27, '09) Chemical Engineer, 86 Norwood Ave., Buffalo, N. Y.
WILKINS, E. M. (Sept. 26, '08) Apartado Postal 54, San Luis Potosi, Mexico.
WILKINSON, Theodore K. (Jan. 29, '09) Balt. Copper Smelting & Rolling Co., Station F, Baltimore, Md.
WILLARD, F. H. (Sept. 26, '08) Commercial Agt., Rochester Ry. & Lt. Co., Rochester, N. Y.
WILLARD, H. H. (Feb. 4, '04) 803 Munroe St., Ann Arbor, Mich.
WILLEY, Leland M. (June 29, '07) Research Lab. Gen. Elec. Co., 230 Liberty St., Schenectady, N. Y.
WILLIAMS, Arthur (Sept. 26, '08) Vice-Pres. and Gen. Mgr., Union El. Lt. & P. Co., 10th and Charles Sts., St. Louis, Mo.
WILLIAMS, H. J. (Mar. 3, '06) 5019 Franklin St., Frankford, Philadelphia, Pa.
WILLIAMS, John T. (Apr. 2, '04) 7 E. 67th St., New York City.
WILLIAMSON, A. M. (Nov. 26, '07) Chemist, with Inter. Acheson Graphite Co., Niagara Falls, N. Y.
WILLS, Henry Le V. (Sept. 26, '08) El. Eng. and Mgr. Savannah Lighting Co.; res., 18 W. Oglethorp Ave., Savannah, Ga.
WILLS, J. Lainson (Apr. 7, '06) Vice-Pres. and Dir. Nat. Brewer's Academy and Consult. Bureau, 400 W. 23d St., New York; res., 133 Midwood St., Brooklyn, N. Y.
WILSON, J. Rattray (Jan. 29, '09) El. Eng., Niagara Falls, N. Y. (Box 801).
WILSON, William L. (Oct. 29, '08) Salesman, Aluminum Co. of America, 158 Lake St., Chicago, Ill.
WINSHIP, Dr. Walter E. (Aug. 5, '05) Elec. Eng., Gould Stor. Bat. Co.; res., 82 W. Washington Place, New York.
WINSLOW, Fred. E. (Feb. 27, '09) Elec. Engr., Iowa Telephone Co., 815 Clinton Ave., Des Moines, Iowa.
WINSNES, Johann (Dec. 27, '07) Eng. Vadheim Elektrochem. Fabrik, Vadheim, Norway.
WITHERSPOON, R. A. (Oct. 2, '02) Shawenegan Carbide Co., Shawenegan Falls, Quebec, Canada.
WITHERSPOON, T. A. (Apr. 3, '02) Ouray Bldg., Washington, D. C.
WITTNEBEL, Ernest S. (Oct. 1, '04) Chemist, A. Klipstein & Co., 122 Pearl St., New York.
WITHROW, James R. (May 29, '09) Associate Prof. of Chemistry, Ohio State University, Columbus, Ohio.
WOHLWILL, Dr. Heinrich (June 29, '07) Norddeutsche Affinerie, Elbstr. 35, Hamburg, Germany.
WOLCOTT, E. R. (Aug. 3, '06) The Amer. Nitrogen Co., Room 623, 108 La Salle St., Chicago, Ill.
WOLCOTT, Townsend (Apr. 3, '02) 39 Whitehall St., New York.
WOLF, Harry J. (Sept. 7, '06) 1748 Welton St., Denver, Colo.

WOLFF, Dr. Frank A. (May 7, '04) Asst. Physicist Nat. Bureau of Standards, Prof. of Elec. Eng., George Washington Univ., Washington, D. C.
WOLFF, R. H. (Apr. 24, '09) Electrometallurgist, Heroult Furnace Co., 445 Broadway, New York City.
WOOD, E. F. (Apr. 3, '02) Vice-Pres. Int. Nickel Co., 43 Exchange Pl., New York.
WOOD, Franklin P. (Feb. 27, '09) Supt. The Black Hills Transaction Co., Deadwood, S. D.
WOOD, Prof. H. P. (Jan. 5, '97) Prof. Elec. Eng., Georgia Technical School, Atlanta, Ga.
WOODWARD, J. M. (May 6, '05) 1228 Citizens' Bldg.; res., 1193 E. 99th St., Cleveland, Ohio.
WOODWELL, A. H. (Oct. 29, '09) P. O. Box 1720, Spokane, Wash.
WOODWORTH, Albert J. (Apr. 24, '09) Electrician, Maryland Steel Co., Sparrows Point, Md.; mailing address, C St.
WOOLDRIDGE, W. J. (Nov. 21, '08) El. Eng., Gen. El. Co., P. O. Box 363, Pittsfield, Mass.
WRAPE, Harold J. (Sept. 25, '09) Secretary, American Carbon & Battery Co., East St. Louis, Ill.; P. O. Box 248.
WRAY, Edward (Jan. 7, '05) 960 Monadnock Bldg., Chicago, Ill.
WURTH, Albert F. (Mar. 27, '09) Mgr., Mold Making Dept., National Phonograph Co., Orange, N. J.; mailing address, 177 High St.
YARDLEY, John L. (Sept. 26, '08) 784 Ellicott Square, Buffalo, N. Y.
YOE, Harry A. (Feb. 27, '09) Testing Dept., Genl. Elec. Co., Schenectady, N. Y.; res., 308 Lexington Ave., Syracuse, N. Y.
YONGE, Allen M. (Sept. 25, '09) Genl. Supt., Aguacate Mines Co., San Mateo, Costa Rica, Central America.
YOSHIKAWA, Prof. Kamejiro (Dec. 27, '07) Imp. Univ., Kyoto, Japan.
YUNCK, John A. (Apr. 30, '08) Glass Expert, Gen. El. Co., Harrison, N. J.
ZAREMBA, Edward (Jan. 28, '08) Mgr. American Fdy. & Mach. Co., 1240 Monadnock Bldg., Chicago, Ill.
ZIMMERMAN, Dr. Fritz (June 29, '07) Chemist, Baker & Co.; res., 147 Orchard St., Newark, N. J.
ZIMMERMAN, J. G. (Nov. 6, '02) 622 Otjen St., Milwaukee, Wis.

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Johnson, W. McA.

Middletown—
Turrentine, J. W.

New Haven—
Bradley, W. M.

Stamford—
Walker, C. R.
Torrington—
Bassett, W. H.
Bradley, J. C.
Waterbury—
Bassett, W. H.
Bradley, J. C.
Bristol, W. A.

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Du Pont, Pierre S.
Weedon, W. S.
Whitten, Wm. M.

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Jenkins, C. Francis
Lloyd, M. G.
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Converse, W. A.
Dryer, E.
Folk, G. E.
Greene, A. E.
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Wilson, W. L.
Wolcott, E. R.
Zaremba, E.
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Chase, M. F.
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Long, G. E.
Wrape, H. P.
Elgin—
Hatch, I.
Granite City—
Mills, J. W.
La Salle—
Lihme, C. B.
Oak Park—
Weld, H. K.
Rockford—
Burson, W. W.
Urbana—
Brooks, M.
Parr, S. W.
Waukegan—
Burling, B. B.
Winthrop Harbor—
Aiken, R. H.

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Bourbon—
Acker, O. E.
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Roush, G. A.
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Bixby, G. L.
Hadley, A. N.
Marti, W. C.
Pumpelly, Jas. K.
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Duncan, T.
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Topping, A. N.

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Winslow, F. E.

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Nutter, C. E.

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Clarke, F. E.

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Bragdon, W. B.

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Larchar, A. B.

Lewiston—
Robinson, A.

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Baker, H. A.
Body, J. F.
Geib, W. V.
Johns, John
Rouse, E. R., Jr.
Smith, E. A. C.
Wilkinson, T. K.

Sparrows Point—
Martin, S. S.
Weymouth, F. A.
Woodworth, A. J.

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Howard, S. F.

Arlington—
Barker, E. R.

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Buchanan, L. B.
Claflin, A. A.
Clapp, E. H.
Goddard, C. M.
Goodwin, H. M.
Hirsch, A.
Jackson, D. C.
Little, A. D.
MacGregor, F. S.
Marsh, G. E.
Melcher, A. C.
Pratt, F. S.
Randall, Merle
Schlesinger, B. E.
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Skinner, H. J.
Stone, C. A.
Stone, J. S.
Talbot, H. P.
Thompson, M. de K., Jr.
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Whiting, J.

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Howard, H.
Lincoln, E. S.

South Boston—
Ruppel, H. E. K.

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Adams, C. A.
Adams, I.
Kennelly, A. E.
Nickerson, W. E.
Richards, T. W.
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Dennison, C. H.

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Coggeshall, G. W.

Fitchburg—
Hayes, C. R.

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Stanley, W.

Lawrence—
Alden, J.

Lynn—
Abbott, W. J.
Berry, E. R.
Heath, H. E.

Malden—
Berry, E. R.
Sargent, F. C.

Peabody—
Manahan, P. R.

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Kelley, J. F.
Unger, M.
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Pickering, O. W.

Swampscott—
Fleming, R.
Thompson, E.

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Weintraub, E.

Winchester—
Tufts, J. L.

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Stanton, F. McM.

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Davies, M. L.

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Marsh, A. L.
Shattuck, A. F.
Stewart, R. S.

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Dow, H. H.
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Wyandotte—
Gibbs, A. E.
Vorce, L. D.

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Morey, G., Jr.
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Lamb, A. B.

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Gradolph, William F.
Hinrichs, Gustavus
Kohler, H. L.
Queeny, J. F.
Williams, A.

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Pearce, John H.
Divide—
Wells, Roy
Great Falls—
Burns, W. T.
Krejei, Milo W.
Wheeler, A. E.
Helena—
Burwell, A. W.
Gerry, M. H., Jr.
Hauser, S. T., Jr.
Hill, S. T. H.

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Omaha—
Millener, F. H.

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Lovelock—
Reid, John T.
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Joyce, C. M.
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Hungerford, Oliver T.
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Myers, Ralph E.
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Seabury, R. W.
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Benoliel, S. D.
Chrome—
Addicks, L.
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Pyne, F. R.
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Rowand, L. G.
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Hart, L. O.
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Davis, Robert W., Jr.
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Highbridge—
Le Boutillier, C.
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Ganz, A. F.
Stillman, T. B.
Irvington—
Cito, C. C.
Maurer—
Colcord, F. F.
Emanuel, L. V.
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Crane, F. D.
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Wesson, D.
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Zimmermann, F.
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Voorhees, L. A.
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Bailey, Frank
Dill, C.
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Roessler, F.
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Caldwell, E.
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Hulett, G. A.
McClenahan, H.
Rahway—
Murray, B. L.
Riverton—
Evans, Chas.
Rutherford—
Daft, Leo
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Sewaren—
Carrier, C. F., Jr.
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Yunck, J. A.
Trenton—
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Watchung—
Moldenke, Richard
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Saunders, A. T.
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Duschak, L. H.
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Nash, A. D.
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Bijur, J.
Block, W. S.
Bogue, C. J.
Bowman, W.
Bradley, C. S.
Bradley, W. E. F.
Breneman, A. A.
Brown, H. P.
Brown, J. S.
Buck, H. W.
Buck, L. H.
Caldwell, E.
Cameron, W. S.
Carse, D. B.
Cary, E. E.
Case, W. E.
Chandler, C. F.
Clark, W. G.
Clark, W. J.
Cleaves, M. A.
Coho, H. B.
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Douglas, James
Dreyfus, W.
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Eimer, A.
Elliott, A. H.
Englehard, C.
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Fitz Gibbon, R.
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Frank, K. G.
Frasch, H. A.
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Gaines, R. H.
Goepel, C. P.
Granbery, J. H.
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Harris, J. W.
Hart, C. D.
Hasslacher, J.
Hatzel, J. C.
Hay, J. A.
Hemingway, F.
Hendry, W. F.
Heroult, P. L. T.
Herzog, F. B.
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Hogel, H. A.
Howell, W. S.
Ihlder, J. D.
Jenks, W. J.
Kahn, J.
Katsura, B.
Kennedy, J. J.
Kenyon, O. A.
Kenyon, W. H.
Kern, E. F.
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Klipstein, E. C.
Kohn, M. M.
Kunz, G. F.
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Langton, J.
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Loeb, M.
Love, E. G.
Lovejoy, D. R.
Lucke, H. J.
MacDonald, J. A.

Mailloux, C. O.
Mastick, S. C.
Mayer, F. J.
Maynard, G. W.
Mays, E. A.
McAllister, A. S.
McBerty, F. R.
McKenna, C. F.
McMurtrie, W.
McNeill, R.
Medove, M.
Mershon, R. D.
Merton, R.
Metz, H. A.
Monell, A.
Monrath, G.
Moody, H. R.
Morehead, J. T.
Morgan, J. L. R.
Morrow, J. T.
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Murphy, E. J.
Muschenheim, F. A.
Nichols, W. H.
Nichols, W. S.
Nodell, W. L.
Parker, H. C.
Pattison, F. A.
Pearson, F. S.
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Price, E. F.
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Randolph, E.
Rawlins, J. A.
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Riker, J. J.
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Roberts, I. L.
Roeber, E. F.
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Rynard, W. T.
Schloss, J. A.
Schuetz, F. F.
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Niagara Falls—

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Acheson, E. G., Jr.
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Arison, W. H.
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Becket, F. M.
Bennie, P. McN.
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Converse, V. G.
Cox, G. E.
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Graham, J.
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Harper, J. L.
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Hepburn, D. M.
Horry, W. S.
Hooker, A. H.
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Marshall, J. G.
Mauran, M.
Meredith, W. F.
Moritz, C. H.
Moyer, G. C.
Ornstein, G.
Petitnot, N.
Phillips, R.
Randall, J. W. H.
Rayner, G. R.
Reilly, E. I.
Reeve, A. G.
Rodgers, A. G.
Saunders, L. E.
Sergeant, E. M.
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Smith, E. S.
Smith, W. A.
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Johnston, W. A.
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Capp, J. A.
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Hansen, C. A.
Langmuir, I.
Lindsay, C. F.
Reist, H. G.
Rushmore, D. B.
Scott, A. P.
Seede, J. A.
Steinmetz, C. P.
Whitney, W. R.
Willey, L. M.
Syracuse—
Matthews, J. A.
Pennock, J. D.
Scott, W. H.
Sykes, H. W.
Wheeler, F. B.
Yoe, H. A.
Troy—
Betts, A. G.
Lincoln, A. T.
Robb, W. L.
Utica—
Johnson, M. H.
Schiller, F. W.
West New Brighton, S. I.
Ingalls, W. R.
Yonkers—
Baekeland, L.
Harrington, E. I.

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Patterson, A. H.
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West Raleigh—
Browne, W. H.

OHIO.

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Geer, W .C.
Shaw, E. C.
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Cincinnati—
Hutchinson, E. J.
Cleveland—
Brown, J. W.
Clymer, W. R.
Collins, 2d, C. L .
Cowles, A. H.
Crider, J. S.
Forssell, J.
Graves, W. G.
Hudson, A. J.
Hyde, E. P.
Koehler, W.
Lane, H. M.
North, H. B.
Smith, A. W.
Whitlock, E. H.
Woodward, J. M.
Columbus—
Allensworth, H. R.
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Kerr, C. H.
Withrow, J. R.
Dayton—
Cridland, H. C.
Deeds, E. A.
East Cleveland—
Baker, C. E.
Elyria—
Hitt, S. E.
Little, C. A.
Fostoria—
Downes, A. C.
Middletown—
Ahlbrandt, G. F.
Salem—
Davis, D. L.
Toledo—
Clarke, W. A.
Youngstown—
Brinker, H. L.

OREGON.

Cascade Locks—
Ballweg, H.
Eugene—
Shinn, F. L.

PENNSYLVANIA.

Altoona—
Casselberry, H.
Walters, H. E.
Allegheny—
Ober, J. E.
Bethlehem—
Landis, W. S.
Rau, A. G.
Smull, J. G.
Braddock—
Friedlaender, E.
Lewis, J. D.
Coatesville—
Coates, J.
Colwyn—
McConnell, J. Y.
Duquesne—
Reed, R. C.
Easton—
Adamson, G. P.
Gordon, C. McC.
Hart, E.
Gettysburg—
Parsons, L. A.
Grover—
Giles, D. C.

Hatboro—
Buckley, H.
Ingram—
Armor, J. C.
Johnstown—
Parkhurst, C. W.
Lebanon—
Weimer, E. A .
McKeesport—
Clarke, E. B.
Milton—
Jenkins, D. J.
Moore—
Werliin, G.
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James R.
Reese, P. P.
Stevens, R. H.
Wales, S. S.
Oakmont—
Thomas, R. D.
Palmerton—
Koch, S. B.
Philadelphia—
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Breed, G.
Cary, C. R.
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Goldbaum, J. S.
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Kitsee, I.
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Sadtler, S. P.
Sadtler, S. S.
Salom, P. G.
Schamberg, M.
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Smith, E. W.
Smith, W.
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Pittsburgh—
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Hartley, R. H.
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James, J. H.
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Laughlin, H. H.
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Merzbacher, A.
Mueller, H. N.
Orr, R. S.
Osborne, L. A.
Page, G. S.
Pinkerton, A.
Rodman, H.
Schluederberg, C. G.
Scott, C. F.
Slocum, F. L.
Snelling, W. O.
Wiley, B.
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Primos—
Boericke, G.
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Reading—
Heizmann, L. J.
Scranton—
Bryden, C. L.
Sewickley—
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An address delivered by invitation of the Board of Directors at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 28, 1909; President L. H. Baekeland in the Chair.

RECENT ADVANCES IN ELECTROCHEMICAL ANALYSIS.

By Dr. Edgar F. Smith.

Mr. President and Gentlemen:—I am deeply appreciative and profoundly grateful to the Board of Directors of the American Electrochemical Society for the invitation which they extended to me to come and spend a short time with you. I have been conspicuous in the Society, I am frank enough to admit, by my absence. I know, perhaps better than you, what I have missed in consequence thereof; but unfortunately I am merely a teacher, and teachers have duties in my part of the country from eight in the morning until six at night, so that I have never been able to obtain a leave of absence extending over a period of time which I felt would justify me in leaving the confines of our institution sufficiently long to attend the meetings in recent years; but when this last kind invitation of the Board of Directors came to me, I resolved that if I could possibly bring it about, I would come over. I have had a delightful time in the few hours I have been in New York in the renewing of friendships made years ago, and I wish it were in my power to stay longer, and sit down and break bread and really be a brother among you.

Thirty-one years ago, while teaching quantitative analysis to boys, I became dissatisfied with the methods then in use for the determination of copper. You are familiar with them as they are laid down in our text books, and you know from experience the difficulties surrounding them, so I was led to try the suggestion of our dear Dr. Gibbs, no longer with us, namely, to let an electric current act upon the salt of copper that was up for analysis and obtained the metal. I am fond of saying, and I doubt whether anyone cares to contradict me, that the early forms of electro-analysis were those of Dr. Gibbs. I remember how I approached the problem—I recall vividly the misgivings I had as to the value of the electric current. Naturally, I did not know whether it was

going to be strong enough to do me any harm or not, or what was going to happen, but I arranged two bi-chromate cells, capacity unknown, and, as I learned afterwards, this gave rise to an energetic current of electricity. With one pole I connected a platinum spiral, that was my anode. It dipped into a solution of copper sulphate contained in a small platinum crucible, round which crucible was wrapped a bit of copper wire, which was my cathode, and I watched that brisk current—that energetic current —and I soon saw the copper make its appearance upon the inside of the crucible.

Not to occupy your time with so trifling a matter, let me say that it became the method in our laboratory used by the boys in the determination of copper.

Cadmium seemed to be a good deal of an annoyance to us in those days, particularly when we undertook to separate copper from cadmium, and the question came to mind, may not cadmium be determined in the same manner as copper? So we substituted a solution of cadmium salt for the copper salt, and the same process was tried, using, however, not the sulphate of cadmium, but the acetate, and there appeared on the sides of the cathode, gray, crystalline cadmium. This was the first time it was determined with extreme accuracy. I was at first a little afraid of the result—I was only twenty-one years of age at that time, but I was interested in everything chemical, and you may imagine how deeply interested I became in that deposit of cadmium, and the keen pleasure I had when I discovered that cadmium could be estimated in that way. Its separation from copper, however, was still bothersome, but that, too, was realized in a day or two afterward, when from a nitric acid solution containing the two metals, copper was deposited free from cadmium.

I continued practicing the electrolytic method for the determination of metals, going to uranium, and was made happy when that great analyst, Fred A. Genth, Professor of Chemistry in our University, said to me: "One of the difficult problems in analytical chemistry is the satisfactory separation of uranium from the alkalies." I had been working, as I have told you, on mineral chemistry, and had had my own troubles with that separation. I said nothing in reply to what the professor observed, but concluded to ascertain what the current would do with a solution of

some uranium salt, using the double acetate of uranium and potassium, with the current from the two bichromate cells, capacity unknown, not measuring the strength of the current at all, but just proceeding in the simplest way. Uranium was deposited as the hydrated proto-sesquioxide on the cathode. It was not long until I was able to tell the doctor that I could determine uranium electrolytically, and also could separate it from potassium. So he wanted to see this separation of uranium from the alkali metal. He saw it, and then turned over to me a solution without telling anything about the quantity of metal with which he started, giving no idea as to the content of uranium, or sodium, or potassium, and said, "Electrolyze that." It was done, and my happiness was increased when informed, after I had repeated the separation three or four times, that my results agreed most accurately with his, both for the uranium and for the sodium, that a separation was completed in an hour's time, a separation which, by the old method which the Doctor pursued, would have required hours, yes days.

Thirty-one years represent a long period of time. You must remember that I was practically a boy working with the electric current, knowing absolutely nothing about electricity, not measuring the current at all but merely letting it act on chemical compounds. There was no Bunsen Electro Chemical Society then, there was no Faraday Society, there was no such Society as this in our country. There was not one publication in any part of the world which gave its pages wholly to work in this particular field. We did not have electricity in the form we have it today. We could not control it as we do today, and as I look back over those thirty-one years, I see one thing after another has been introduced into this field, in other words, how it has become a distinct branch of chemistry, and while my contributions did not amount to anything very remarkable (though very interesting to me), they were yet contributions. Of course there were many discouragements, they came from everywhere, and I heard many persons remark: "Why does he fritter away his time on that sort of thing? There is nothing to it." Discouraged a bit on hearing such comments, I yet persevered, and went to work on molybdenum and tungsten, to discover what action the current had upon their salts. I sometimes think we follow fashion a little too much. We are a

little bit afraid to walk in the avenue that attracts us for fear of the criticisms which may come. We realize that we are not great chemists. But we do not amount to anything unless we can make our analyses with absolute precision. In spite of all my efforts to get away from the use of the electric current in analysis, I was drawn back every time, and I would like to show you some of the things which have come up at intervals, that have been responsible for these many years of devotion to this extremely narrow field in a corner of the great domain of chemistry.

I have missed much, I have said, in absenting myself from the meetings of the Society. The papers read here this afternoon have been extremely interesting to me, and perhaps the little experiments which we are going to show you this afternoon may bring to the mind of someone present a thought which will be helpful in the field of practical electro-chemistry, and that is another reason for coming.

In the old days of electro-analysis, but few metals were determined by the help of the current—e. g., principally copper and lead. To these, others have been added from time to time. In going about the country, listening to my fellow chemists, I have frequently heard "The objection to the electro-chemical method is that it requires too much time. The current must act all night, twenty-four hours sometimes, and it is an expensive affair. In the end the results really are not any more trustworthy than those obtained by the time-honored ordinary gravimetric procedure. You cannot apply these methods to technical problems. They are all right in analyzing a pure salt, or double salt, but with complicated mixtures, which the man in the practical field of chemistry must face, the electrolytic methods are of no consequence." These are the criticisms.

Tungsten has always been a favorite element with me, as has been molybdenum. Having in mind the separation of the two metals, it was soon learned that it was possible to deposit molybdenum as a hydrated sesquioxide. With a normal tungstate and exposed to the same current conditions, there was no deposition of tungsten, so that their separation in the electrolytic way seemed probable. The molybdenum deposit, however, invariably contained tungsten. On watching this deposition of molybdenum, blue threads were seen passing out from the cathode towards the

anode, and as they approached the latter vanished. The idea then came to mind that if the electrolyte would be agitated, these blue threads, an oxide of tungsten, would not appear at all, there would be no reduction of the tungsten; so the agitation of the electrolyte suggested itself. In 1886, Von Klubukow agitated the electrolyte by rotating first the cathode and then the anode, and later, holding the electrodes stationary, rotated the electrolyte. This was a new idea, this agitation of the electrolyte, and he said, after a great deal of experimentation, that the rotation of the anode was the best. His anode rotated 120 times in a minute, but he never published any results. In 1901, when the molybdenum and tungsten separation was in mind, we hoped that agitation would perhaps enable us to effect this separation, and the further thought arose that with agitation we might increase the current, and the pressure. We did not succeed, as I said, in separating molybdenum from tungsten, but we put into vessels of this kind (exhibit vessel), copper sulphate, murcurous nitrate, etc., and while rotating the anode, agitated the electrolyte, and we were able by turning on a high current under great pressure, to throw these metals out in a few minutes. We dropped the experiments for a while, until 1902, and then resumed them, but never said anything to the public about it. In 1903 we began a systematic study of the metallic salts we had in hand with a view of seeing whether it was possible to deposit them from a number of electrolytes in a short time by agitating the electrolyte, while increasing the current and pressure.

This platinum dish contains a solution of the double cyanide of potassium and copper. There is 0.25 gram of copper present. While agitating the electrolyte by rotating the anode, we propose to pass through it a current of about six amperes, under a voltage of about 16. Chemists may say that double cyanide of potassium and copper is a peculiar electrolyte. Indeed, there are those who tell us that cyanide of potassium will dissolve the platinum from the anode, but this will not occur with the amount of cyanide we have here. Hundreds of experiments have demonstrated this fact. We have carefully examined the electrolyte after it was poured away from the deposit and were not able to find platinum in it, nor is there any platinum in the copper deposit.

Others metals, difficult to determine in the ordinary way, may

be quickly estimated by this scheme. Now, we shall determine molybdenum. It will not be deposited as metal. It comes down on the cathode as hydrated sesquioxide, but it is completely precipitated, and I feel sure those of you who have molybdenum to determine will be willing to give the suggestion a trial. We use it altogether. It has been used abroad in a number of laboratories, and the results have been sent to us. They show a satisfactory outcome.

I know that the determination of zinc in an electrolytic way has been discussed before this Society quite a bit. Zinc is one of the metals which the analyst is apt to meet frequently. We are throwing it out from solution in about ten minutes—a quarter of a gram. We propose, however, to change the cathode in this case (illustrating). In stead of using a platinum cathode we shall use a nickel cathode. The zinc is difficult to remove from the platinum when it once comes in contact with it, but from such a cathode as this (nickel) we shall have no trouble. Notice the character of the deposit. This electrolyte is sodium zincate. We have had just as good success using the acetate as an electrolyte. If you were to go to Philadelphia you would find the students in our laboratory at work every day, using methods of this kind, and one thing which I think would strike you is their intentness upon the work.

Please notice the character of the anodes. There is a flat spiral. Sometimes to hasten matters, we use an anode like this, (illustrating). This is a dish of the same shape as the cathode. When that is used as an anode, rotating 500 or 600 revolutions a minute, it is possible to throw down a half gram of palladium in two minutes, and a half gram of silver in five minutes. I fancy that some of you will say that this scheme is expensive, that the cost of the platinum wire and the dishes is considerable. Well, wait just a moment, and we shall try to offer a substitute for this expensive outfit, although in every laboratory where a great deal of work is to be done, the matter of a few platinum dishes is a small item, when it is important that determinations should be made quickly and correctly. Here is a salt of sodium molybdate acidulated with a drop or two of sulphuric acid, and the current turned on as you observe. In this case twenty minutes will be required to throw down a quarter of a gram of molybdenum in

the electrolytic way. This would require an hour or two with the method formerly in use, and I am sure under these circumstances, everyone would prefer the electrolytic way. Bear in mind that this deposit of molybdenum is not metal, it is the oxide, and in carrying out the determination, it is customary after the precipitation is finished, to wash the deposit carefully with warm water, then dissolve in a little nitric acid, evaporate to dryness and ignite gently. There is an indicator here to show us when precipitation is at an end. In the beginning the liquid in the dish becomes blue in color, but the blue disappears as the metal goes out. Antimony may be determined in this way. It separates in a gray metallic form. The best electrolyte for nickel is the double sulphate of nickel and ammonium.

Let what I have said be the first paragraph of my talk and we shall proceed now to the second paragraph. In 1880, Wolcott Gibbs placed a solution of copper sulphate in a beaker containing mercury. This platinum (exhibiting specimen) runs down into mercury, which is so connected that the mercury becomes cathode, and a second platinum wire, running into a spiral at the end, is made the anode. By using the same energetic or brisk currents, Gibbs found it possible to precipitate out the copper from the solution completely. Of course, the sulphuric acid of the salt remained in the water. Gibbs gave no quantitative results. He merely said that a copper sulphate solution, when electrolyzed in this manner, lost color. Later a little work was done on iron phosphate dissolved in sulphuric acid, the iron being completely deposited in the mercury, while the phosphoric acid remained in the solution. In our laboratory we undertook to repeat this work of Gibbs, to learn whether it was quantitative, whether the idea could be made helpful in quantitative analysis. After a time we discovered it was not well to have the platinum wire running into the mercury in order to make it the cathode, because in the precipitation of copper, for example, there would frequently be found on the platinum wire near the mercury, crystals of copper, and to prevent their loss it was necessary to weigh the platinum wire, along with the beaker and mercury. We substituted a pencil of carbon for the platinum wire, and afterwards this little device (exhibiting device) put up by one of the students of the laboratory. It is nothing more than a test tube with a flat bottom

through the side of which a piece of platinum wire is fused. Standing it upon a plate of copper like that (illustrating) it becomes the cathode. The copper sulphate is next introduced into this cup, which has been weighed and contains about twenty grams or more of mercury, and is then ready for decomposition. In a short time after the current has been turned on, it is possible to weigh the copper. We determine zinc, iron, cobalt and a number of other metals by this method. We do not require the expensive platinum dish. This little cup will cost perhaps not over twenty cents. Anyone can make it who is good at glass blowing. We electrolyzed copper sulphate, chemically pure, deposited the metal in the mercury, and then determined the sulphuric acid in the residual liquid by titration. Those of you, who in your students days analyzed copper sulphate by precipitating the copper by caustic alkali, know the trouble experienced in washing the precipitate and how long it took to get the sulphuric acid—and all this may be done in ten to fifteen minutes. The separation of iron and titanium, ordinarily difficult, may be made in this way. The iron is deposited in the mercury; the titanium remains unprecipitated.

Follow me a little further, please, with this cup. We have here nickel sulphate. It is being electrolyzed with a current of five amperes and a pressure of twelve volts. Those of you who are watching this, noticed the color at the beginning—you see the green is disappearing, which indicates that the nickel is being precipitated. Wash the amalgam with water, with absolute alcohol, and then with ether, and let it stand in a desiccator for ten or fifteen minutes and weigh it. The determinations are really very accurate. This system we have been using right along in our laboratory. All it consists of is this platinum rod, wound into a spiral and the little cup with the platinum fused in it. You will be surprised at the excellent results obtained by neophytes, so to speak, when working with the various metals.

Suppose chlorides are electrolyzed, what then? Take cobalt chloride—the chlorine is not wanted, but when it is dissolved perhaps the platinum will be affected. Do you know how we offset that? We place above the metallic solution a layer of toluene, and as the chlorine is liberated, it will be promptly absorbed by the

toluene. We have analyzed cadmium bromide in such a cup as that, and not a particle of bromine was apparent.

There is another thought and it is this—you know our electro-analyses thus far have been with the heavy metals. When we got to the alkaline earth metals the literature was silent. This was, also, true of sodium or potassium, but we now can speak of them. Let me tell you that story as briefly as possible. Can we electrolyze this solution of sodium chloride? We will not use a platinum but a silver anode. Why? Because then the chlorine ions may rest upon it, and that is what occurs. We coated a platinum anode with silver. Let us call it a silver anode. It was first made of a bit of foil. The sodium went into the mercury, of course, and the chlorine went over to the silver anode. The silver anode was weighed before and after, and its increase in weight gave the theoretical amount of chlorine. Just as soon as the needle on the ammeter fell back to zero, we no longer had any salt in solution so we took out the silver anode, washed and dried it. Then, what did we do with the other part, the amalgam? We covered it with water, dropped in a platinum anode, reversed the current and pushed the sodium out into the water. There is an analysis of common salt made in thirty-five minutes! (Applause). We also analyzed barium chloride. This is not far away from your student days. That is, in many laboratories, probably the first example given in this salt. The barium and the chlorine are to be determined and we who are teachers know the results and the time required. With this little scheme the barium chloride solution is thrown into the cup, mechanically agitated, in conjunction with the mercury. In less than an hour, results are obtained such as no beginner ever meets in the ordinary analysis of barium chloride. After a while, we noticed that the anode of silver chloride became brown. It did not take long to discover that some of the amalgam was decomposing, because the current had run a little too long. Caustic came in contact with the chloride and formed an oxide, so that sometimes we had a little more on the anode than we wanted. We tried all sorts of schemes to obviate this. We took a crystallizing dish (like this) and within it suspended a bottomless beaker. In the inner cup was introduced the sodium chloride solution; in it was suspended the anode. The mercury is made cathode by the platinum wire. When the

current was turned on there was, of course, no trouble. We do one thing more. We rotate the anode. Then the sodium amalgam comes outside, meets the water, and is there decomposed into sodium hydrate. Instead of a vertical plate of platinum or silver as anode, we substituted a gauze disc of platinum, coated with silver.

I am not much of an ionist. Our friend Bancroft says I am a physical chemist, but I do not think so. I am just an ordinary chemist and not much of an ionist, but listen to this story. It is on me, of course. A young man working with this particular form of apparatus while making these determinations—he doing the work, and he and I talking it over together—came to me one day and said, "Don't you think you ought to be converted now when we can send these anions here and the sodium ions there, don't you think you should begin to believe in electrolytic dissociation?" I said, "Suppose you put sodium carbonate in that inner cup, and if the CO_3 comes over here to the silver anode, then I will be a convert." Do you know, it went! It just did that thing! It went over there. We had weighed the silver anode, and then we dried the silver anode, after the experiment, and re-weighed it. The result appeared in the increased weight of the anode. There is no question about it, that ion, CO_3, went over to the silver anode. The anion, PO_4, went over there. They hung themselves up on the silver and allowed themselves to be weighed.

To me, all this was most interesting, and while I was trying to get away from electrochemical analysis to work of another character, these things kept pulling me back. I had to come back, I did not want to but they brought me back, so that I have come to be just a bit enthusiastic on this subject. I do not know whether there are ions or not. I know, however, that we can weigh these acid groups. We determine the alkali that comes out as accurately as any one here in this audience can ask it to be determined.

Let us next electrolyze sodium chloride—common salt. The chloride will be decomposed. After a moment the amalgam will come outside and decompose, it is swished out, and you see the hydrogen bubbles rising. The inner cup is held in a vertical position with rubber stoppers. Chlorine, bromine, iodine and numerous other negative groups are thus determined. Ammonium chloride has been decomposed in this cup. To my mind, there is such a thing

as NH_4-amalgam; it is fairly stable but decomposes quickly with water. The negative groups have all been caught up by silver and held. When we electrolyzed sodium tungstate in this double cup we at first met with little success. Often one works for months, yes years, before one gets any kind of thought that is worth while, so at length there came the idea that perhaps better results might come if we put a diaphragm under the inner cup. We then electrolyzed sodium tungstate using a lead anode; the tungstic acid immediately attached itself to the latter. We also electrolyzed fluorides in this double cup arrangement. In this case a platinum gauze anode was coated with calcium hydrate. Calcium fluoride was precipitated on the anode.

The use of horizontal discs as anodes led to their multiplication. Here is one with six such discs upon it, and you may ask me why we multiply the number of discs. The thought came to us that we might determine the atomic weight of sodium or chlorine by decomposing sodium chloride in this cup. Mr. Goldbaum, who is here with me, carried out about half a dozen determinations of the atomic weight of sodium in this way, starting with pure sodium chloride, and gathering more than a gram of chlorine on the discs.

You are all interested in radium—chemists cannot help being interested in it, but the trouble is to free radium from barium. In our laboratory, while mulling over these things, it occurred to us that possibly if we had some radiferous barium chloride, we could separate the barium. Dr. Baskerville sent me 1/200 of a gram of pure radiferous barium chloride. He told me the activity, and we tested it over there, and we separated the radium from the barium. Here is the identical piece of apparatus (illustrating) in which the separation was made. You observe the crystallizing dish is not so large. This is the silver anode—platinum disc coated with silver. You can see the decomposition on the outside, and the liquid siphoning off from the outside compartment will, when it is evaporated, have an enormous effect upon the gold-leaf electroscope.

Is it possible for us to separate potassium from sodium, if we had the chlorides? Yes, it is. Those who make the usual analysis with a little potassium and a little sodium, and have trouble with the separation of chlorides, are recommended to try this method.

You can use this apparatus and have the benefit of our experience. We know from the experience we have had that it is easy of execution.

These are just a few of the novelties, or a few of the points in modern electrochemical analysis, which, at the request of the Board of Directors of this Society I have the pleasure of laying before you. You recognize how attractive the field is. It is but one little corner in the great field of electro-chemistry. It holds some of us there, in spite of our best efforts to get away from it. I am so enthusiastic on this subject, that I believe the day is coming when we shall analyze a great many minerals in the electrolytic way. If you ask me how we are going to do it, I do not know, but we are going ahead and shall try to do it. What the method will be no one knows, but something will come. When I was a student, if anyone had said we were going to analyze common salt in this fashion, in thirty-five minutes, we would have thought it incredible, and yet it can be done.

Mr. Goldbaum can bear witness to the fact that boys who have had quantitative analysis for the first year—seniors now— come into the laboratory and make these determinations. We have a record of them and will lay them before you later. Anyone is welcome to come, examine and see for himself. If you are skeptical, the quickest way to remove the doubt is to accept our cordial invitation to come to Philadelphia and see the evidences of these determinations for yourselves.

DISCUSSION.

Prof. Henry E. Armstrong, of London, England: Mr. President and Gentlemen. I count myself very happy in having come here today and had the opportunity of listening to the charming account given by Professor Smith of his remarkable work. Of course, every chemist has become acquainted with Professor Smith's work, but, after all, the ideas we get when we read about these things are very feeble, indeed, in comparison with the impressions we get when we come face to face with the man and have some opportunity of judging of the way in which

he looks at his work. Professor Smith may or may not be a physical chemist, but it is quite clear that he is a very human person, with a peculiarly characteristic personal equation, and we have had the opportunity of realizing something of the value of that equation, and we can very easily see what an influence such a man must exercise upon his students.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City October 30, 1909, President L. H. Baekeland in the Chair.

A NEW ELECTROLYTIC METHOD FOR THE PREPARATION OF EXPLOSIVE ANTIMONY.

By R. C. Palmer and L. S. Palmer.

Introduction.

Under certain conditions antimony is deposited electrolytically from solutions of its halogen salts in such a form as to explode with the liberation of antimony trihalide fumes. This so-called explosive antimony was discovered by Gore[1] in 1859, when he electrolysed a hydrochloric acid solution of antimony trichloride, with antimony and copper as anode and cathode, respectively. In his paper, Gore pointed out that the formation of this unusual deposit depended on the concentration of the electrolyte, the current density and the temperature.

Gore's investigation was followed by a number of others, that of Cohen[2] being the most important. He showed conclusively that the antimony trichloride contained in the deposit was united chemically with the metallic antimony of the deposit, forming a solid solution of amorphous antimony and antimony trichloride, more antimony being deposited than is required by Faraday's law. He also noted, as did Gore, that a small amount of hydrogen chloride was held in the deposit mechanically. Cohen also determined the exact experimental conditions which favored the formation of the explosive deposit. They were (1) concentration of electrolyte greater than 10 per cent, (2) temperature less than 15 degrees C., (3) high current density, *i. e.*, 0.043 to 0.43 amperes per sq. cm,, (4) vigorous stirring of the solution. Cohen also prepared explosive antimony from an alcoholic solution of antimony trichloride saturated with hydrogen chloride.

[1] Phil. Trans. Roy. Soc., London, 1858, p. 185.
Phil. Trans. Roy. Soc., London, 1859, p. 797.
Phil. Trans. Roy. Soc., London, 1862, p. 323.

[2] Zeit. Phys. Chem., **47**, 1904.
Zeit, Phys. Chem., **50**, 1904.
Zeit. Phys. Chem., **52**, 1905.

Preliminary Investigations.

In the course of the preparation of some explosive antimony, it occurred to us that this deposit might be formed by the electrolysis of other non-aqueous solutions of antimony trichloride. A number of solvents for antimony trichloride were tried. Liquid sulphur dioxide, nitro-benzine, formic acid and acetone proved to be excellent solvents and yielded excellent conducting solutions. Acetone seemed most favorable, so we chose that solvent to carry out our investigations.

Some interesting experiments with liquid sulphur dioxide and nitro-benzine were carried out before investigating acetone. We did not succeed, however, in obtaining the explosive antimony from solutions in these solvents.

We took an average solution (about 20 to 30 per cent.), kept it at about 0° C., and set out to determine what current density would give explosive antimony. As the result of several experiments we found that high current densities, like those used by Cohen, would not give explosive antimony from acetone solutions free from hydrogen chloride, but by employing much lower current densities, explosive antimony was obtained.

Plan of Work.

Having obtained explosive antimony from acetone solutions, and having determined the most favorable current density for its formation, the object of this investigation was primarily to ascertain the effect of the concentration of the electrolyte on the character and composition of the deposit, no hydrogen chloride being present in the solution.

Using the purest materials obtainable, samples of explosive antimony were deposited electrolytically from solutions ranging in concentration from 10 per cent. to 75 per cent., at a temperature of about —4° C., using a current density of 0.0058 amperes per sq. cm. of cathode surface. The anode was a cast bar of pure antimony, the cathode a platinum cylinder, and the solution was vigorously stirred during the deposition. The electrolytic cell was closed to exclude moisture. About 20 to 25 hours were required for the preparation of each sample.

The samples thus obtained were then analyzed. They were exploded by heating in an atmosphere of dry carbon dioxide in closed tubes. After driving off all the antimony trichloride contained in the deposit, the expelled salt was dissolved in ether-

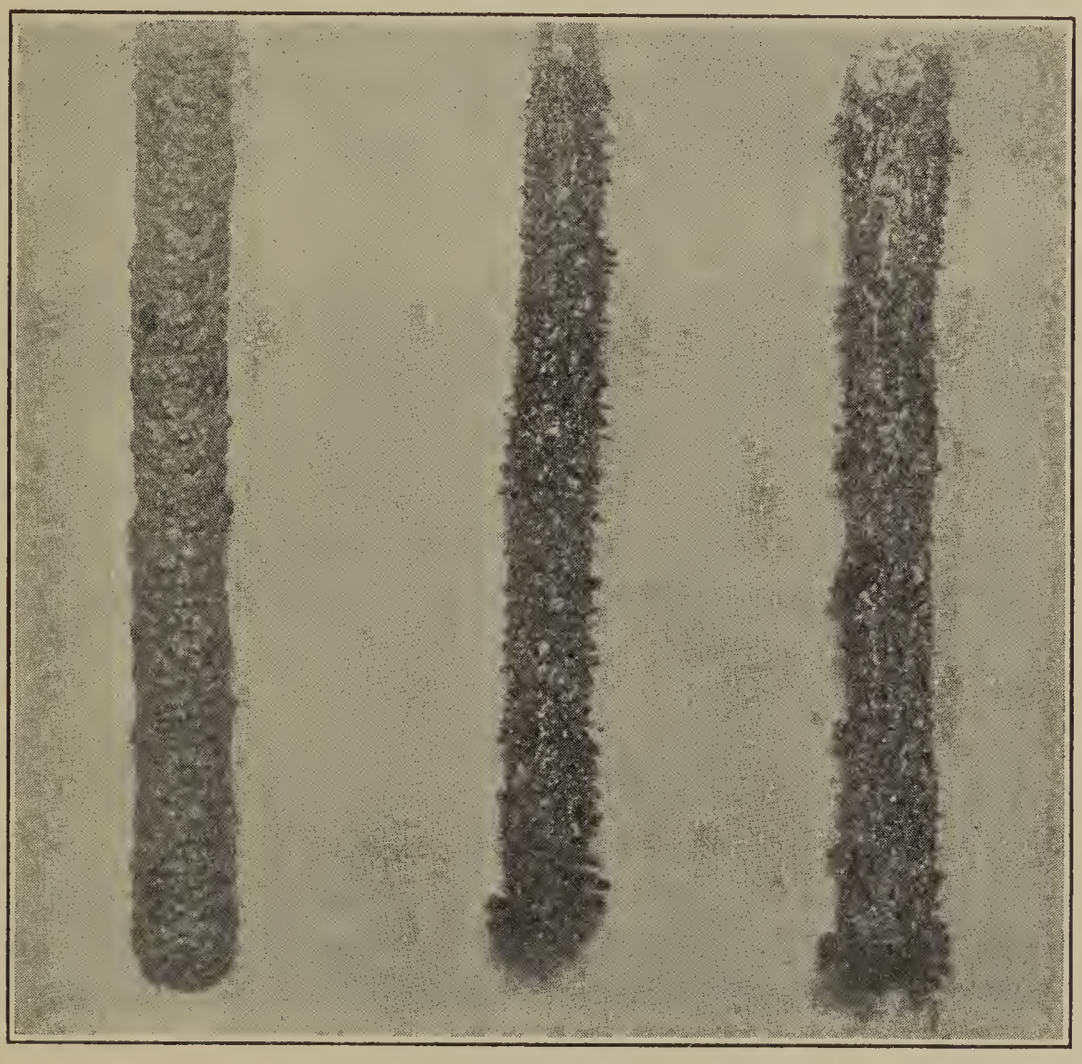


FIG. 1.

alcohol and the solution analyzed electrolytically for antimony, using sodium sulphite to reduce the polysulphides as mentioned by Smith[3] and Fisher.[4] From this weight of antimony the percentage of antimony trichloride in the deposit was calculated.

RESULTS.

The character of the deposits obtained was very different from those obtained from the usual hydrochloric acid solution. This

[3] Electrochemical Analysis, E. F. Smith, p. 115-119.
[4] Elektroanalytische Schnellmethoden, Fisher, p. 135.

marked difference is shown clearly in Plate I. Nos. 8 and 9 are from 55 per cent. and 65 per cent. solutions of antimony trichloride in acetone, while No. (A) is a sample prepared from an aqueous hydrogen chloride solution of antimony trichloride. The former, while they have a lustrous appearance, are flaky and not very adherent, although they were deposited on tubes of roughened platinum. The latter has its characteristic knobby appearance, and is hard and adherent. The deposits from the acetone solutions were quite as explosive as those obtained from hydrochloric acid solutions. Simple scratching with a knife blade produced the explosion.

Gore[5] and Cohen[6] both noted a small quantity of mechanically held hydrogen chloride in samples prepared from hydrochloric acid solutions. Similarly we noted that a marked quantity of acetone was liberated when the samples prepared in acetone were exploded. This quantity of acetone increased with increased concentration, being sufficient in the higher concentrations to give a brown color to the liberated antimony trichloride fumes, and to give them a peculiar organic odor. The amount of included acetone was not determined.

The results of analyses, together with some other experimental data, are given in Table I.

An examination of the data will show that with increased concentration of the electrolyte, there is an increase in the percentage of antimony trichloride contained in the explosive deposit. Two results, *i. e.*, from the 15 per cent. and 45 per cent. solutions, are surprisingly low. We are unable to account for this satisfactorily. Probably some metallic antimony was deposited with the explosive antimony, due to some conditions in the electrolysis of which we were not aware.

We wish to call particular attention to sample No. 11 which was prepared in a 30 per cent. antimony trichloride-acetone solution, which had been previously saturated with hydrogen chloride. This sample was quite hard, and resembled very much that prepared in the usual aqueous acid solutions. In this case a current density of 0.112 amperes per sq. cm. was used, which is twenty times that used for acetone solutions free from hydrogen chlo-

[5] *Loc. cit.*
[6] *Loc. cit.*

ride. This point is of special interest. When a current density as high as 0.112 amperes per sq. cm. is used in acetone solutions free from hydrogen chloride, explosive antimony will not be deposited.[7]

TABLE I.

Data.

No. Sample	C. D. Amp./Sq. Cm.	Voltage Volts	Average Temp. °C	Conc. of Elect. Per cent.	Wt. Exp. Sb. Grams	Wt. $SbCl_3$ in Deposit Grams	Per cent. $SbCl_3$ in Deposit	Remarks
1	0.0058	11	—2	10	1.1725	0.0397	3.39	
2	0.0058	8	—3	15	0.9655	0.0148	1.54	
3	0.0058	7	—4	20	1.2278	0.0613	4 99	Slightly treed dep., bright underneath.
4	0.0058	6	—5	25	0.9724	0.0581	5.97	
5	0.0058	5	—5	30	1.6195	0.0856	5.29	
6(*a*)	0.0058	4.5	—5	35	2.0836	0.1304	6.27	
7	0.0058	3.5	—5	45	1.5539	0.0611	3 94	
8	0.0058	3.25	—4	55	1.8215	0.1385	7.62	Few trees, flaky deposits.
9	0.0058	3	—3	65	1.7549	0.1405	8.02	
10	0.0058	2.75	—2	75	1.3567	0.1178	8.32	No trees, bright flaky deposit.
11(*b*)	0.112	. . .	5	30	1.7134	0.1110	6 48	Hard dep. slightly treed.

(*a*) This is probably a little low, as a small part of the sample exploded when it was put in the heating tube.

(*b*) Sample prepared in acetone solution saturated with hydrogen chloride.

SUMMARY.

1. Explosive antimony can be obtained by electrolysing 10 per cent. to 75 per cent. solutions of antimony trichloride in acetone without the presence of any hydrogen chloride, using a low current density and at a temperature of about 0° C.

2. A small amount of acetone is included in the deposit.

3. The deposit has a characteristic flaky appearance, differing from the deposit obtained in hydrochloric acid solutions.

4. The percentage of antimony trichloride included in the

[7] In Volume XV, Transactions of this society, Patten and Mott, in a paper on "Electro-deposition of Some Metals from Acetone Solution," state that smooth deposits of metallic antimony were obtained from acetone solutions of $SbCl_3$, between platinum plates, using a current density of 0.0007 amps. per sq. cm.

deposit increases with increase of antimony trichloride in the electrolyte, a result which was found by Cohen, using aqueous solutions.

These are the first experiments on record in which explosive antimony was obtained from solutions in which no hydrogen chloride was present.

This work was undertaken at the suggestion of Dr. Herman Schlundt, Professor of Physical Chemistry in the University of Missouri, and we wish to acknowledge his interest and many helpful suggestions.

Chemical Laboratory,
University of Missouri,
October 21, 1909.

A paper read at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909, President L. H. Baekeland in the Chair.

THE PRACTICAL CONDUCTANCE OF ELECTROLYTES.

By Joseph W. Richards and Walter S. Landis.

Under the above title we mean to discuss the actual conductance of the electrolyte in electrolytic refining tanks as the same are ordinarily set up and operated. In almost all refining tanks the edges of the anode and cathode plates, they being usually of the same size, are at some distance from the sides and bottom of the tanks. The free space, that not directly included between the plates, is a field through which stray current flows, the current lines being usually curved. It adds considerably to the conductance of the electrolyte. We have endeavored to measure experimentally the conductance of these lateral or bottom prisms of electrolyte in order to see to what extent they influence the conductance of the electrolyte as a whole.

The experimental method used was to place two electrodes ABCD and EFGH (Fig. 1) of pure copper, in a neutral copper sulphate solution, against the ends of a glass cell which they completely covered (in the figure they are shown raised from the bottom). Readings of current and voltage allowed the calculation of the conductance (= current/voltage) of the cell as a whole, repeated readings being taken at different values of current and voltage until accurate values were obtained for the conductance. The plates were then raised by intervals of one centimeter and the conductance of the cell determined each time as before. From the conductance of the cell as a whole, as thus measured at each interval of rise, was subtracted the calculated conductance of the prism of electrolyte ABCDEFGH directly between the plates, leaving the conductance of the bottom prism ABKLMNGH. The conductance of the bottom prism was then compared with its full calculated conductance, *i. e.*, that which it would possess between opposite parallel plates ABKL and HGMN, and the percentage which the former formed of the latter recorded. Similar

experiments were made with cells of different dimensions. It was found that while the conductance of the bottom prism was different for different lengths between the plates, its percentage conductance as calculated above was practically constant for any given ratio of depth of solution AK under the plates to length of solution AH between the plates. We have, therefore, plotted from our most reliable experiment the percentage conductance

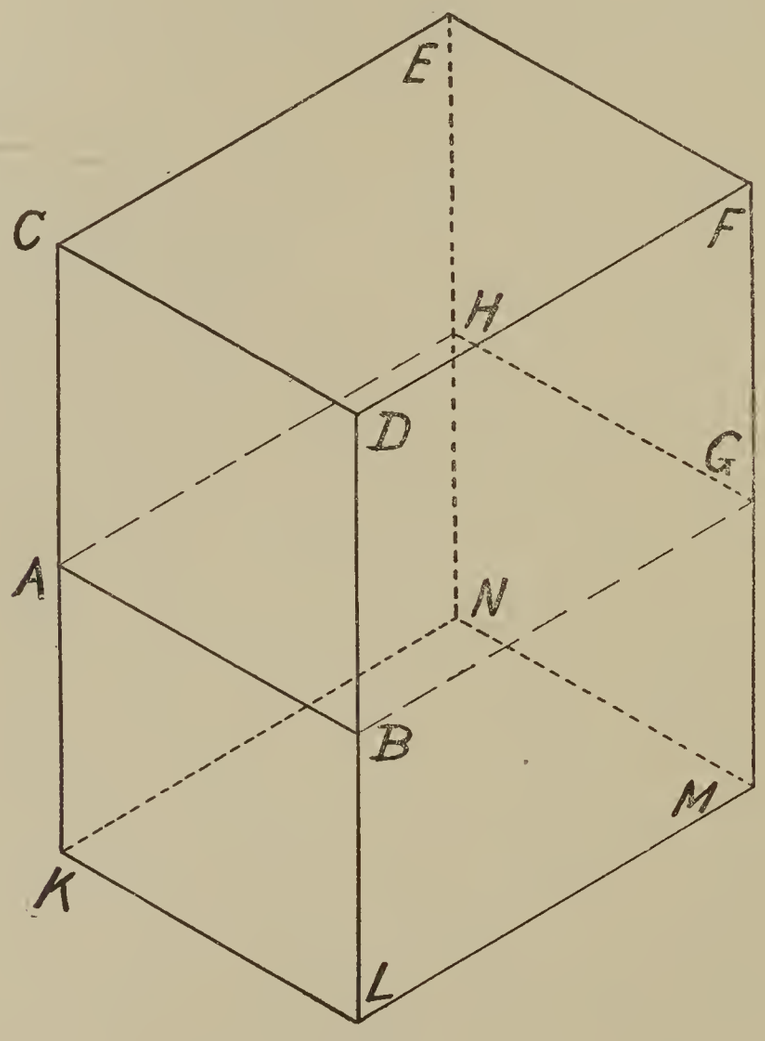


FIG. 1.

of prisms below the plates, taking as ordinates the ratio of depth to length and as abscissas the percentage conductance as calculated above. See Fig. 2.

For zero ratio, the conductance is, of course, 100 per cent., and for an infinite ratio 0. An interesting observation is that for a given conductance between the plates the actual (not percentage) conductance of the prism below the plates reaches a maximum at a finite increase in depth and thereafter remains constant. In several of our experiments this maximum conductance was

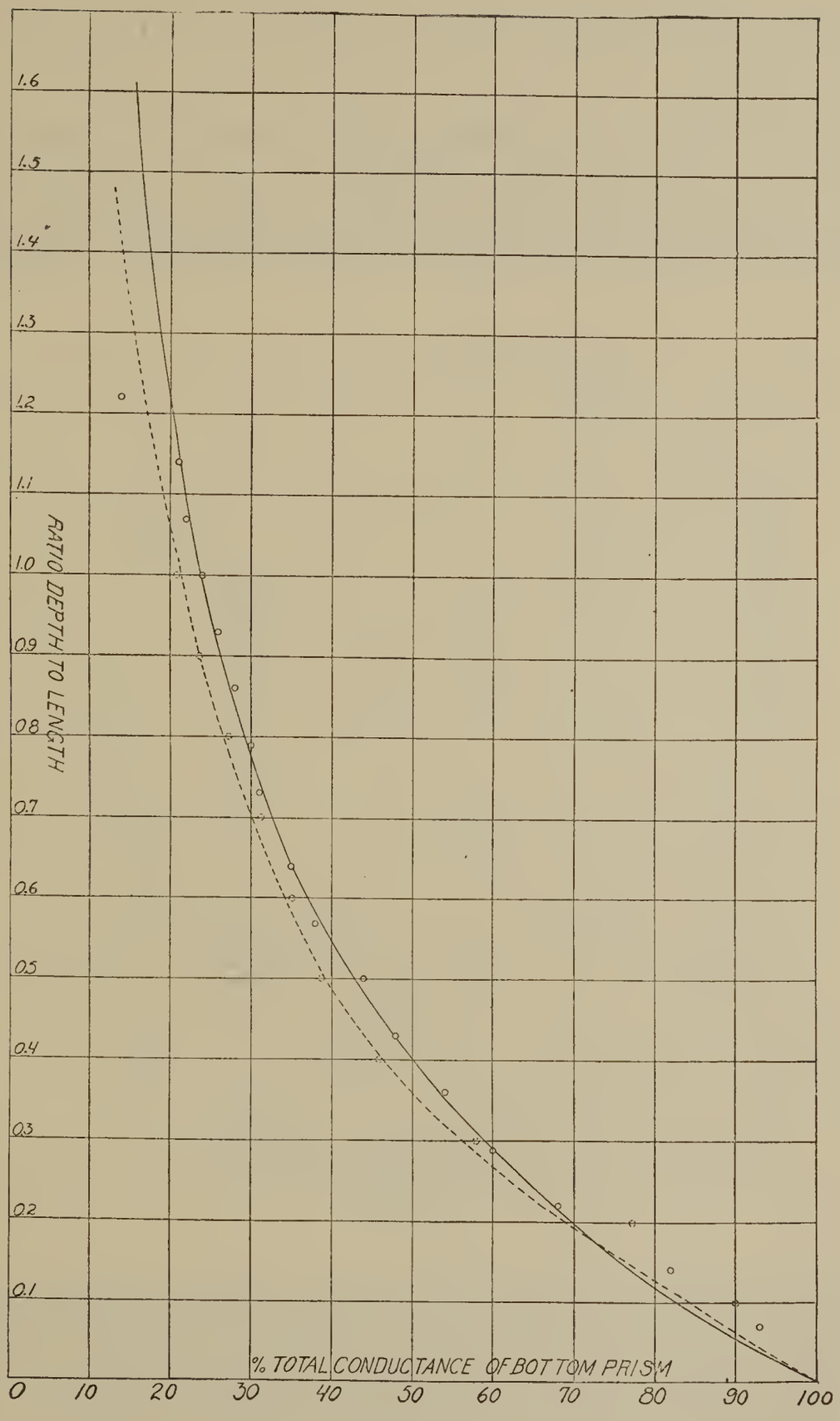


FIG. 2.

reached with a ratio of depth to length of approximately 0.8. We have plotted in Fig. 3 the percentage which the actual conductance bears to the determined maximum for the prism beneath

the plates, the ordinates again representing the ratio of depth to length and the abscissas the percentage of the maximum conductance shown.

In order to determine whether the conductivity (specific conductance) of the electrolyte affected the values determined as above, we tested electrolytes of widely varying conductivity with approximately identical results, and furthermore tested sheets of

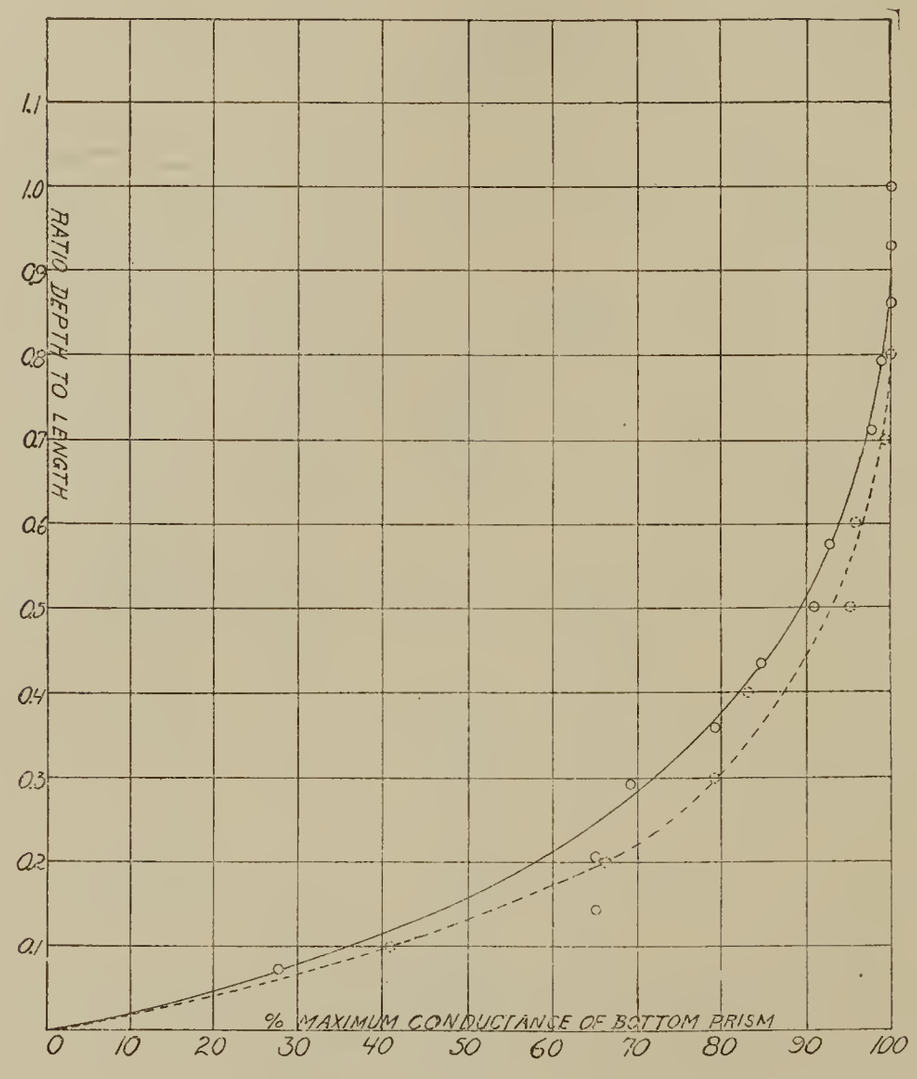


FIG. 3.

copper, tin foil and zinc, the most careful experiments being made with zinc, and the ratio determined from it practically duplicated the ratio obtained from the copper sulphate electrolyte. The results obtained on the zinc are shown in Figs. 2 and 3 by the dotted line.

In cells in which the electrode covered only a portion of the ends, we also attempted to determine the conductance of the prism of electrolyte not directly underneath the electrodes, but to one side,

i. e., as in Fig. 4, the prism CNOKLGPQ, diagonally under the electrodes, and which may be assumed as having connection with the electrodes only at its corners C and G. In making these determinations, electrodes ABCD and EFGH were used smaller than the ends of the cells and were also raised up by successive centimeters, leaving besides the prism ABCDEFGH directly between the plates, the side prism CDKSLGFT between the edges of the

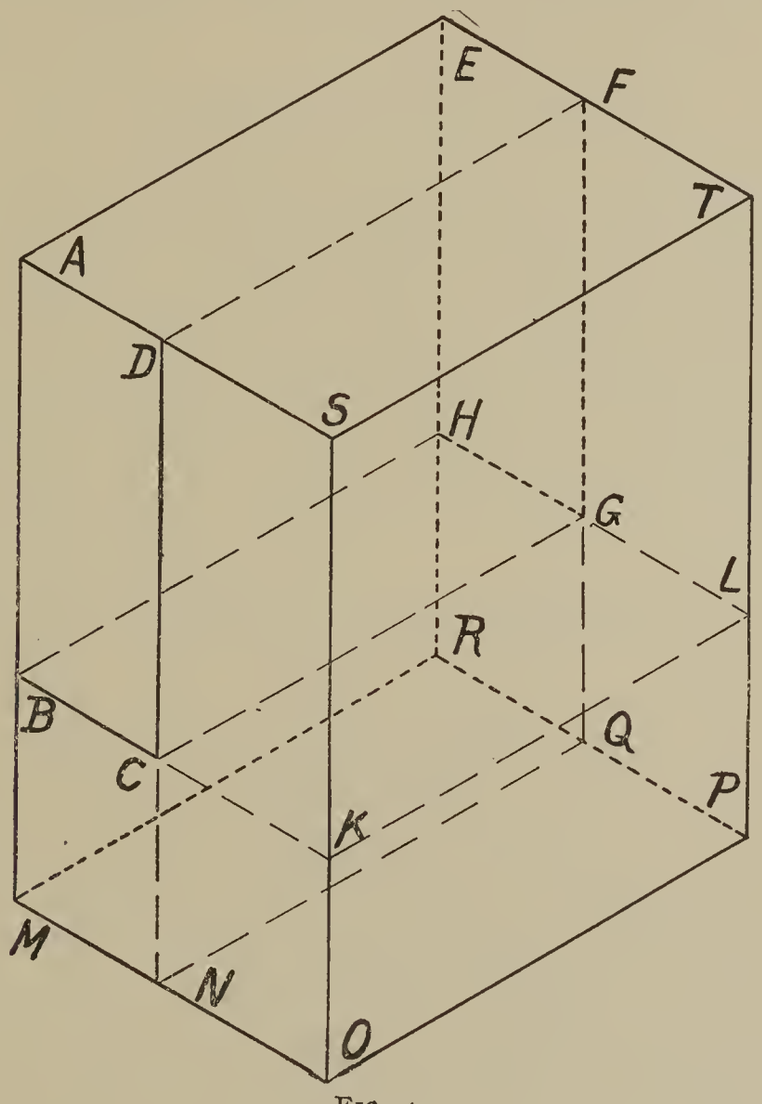


FIG. 4.

plates and the side of the tank, the bottom prism BCMNQRHG between the bottom edges of the plates and the bottom of the tank, and the diagonal bottom prism CNOKLGPQ. The conductance of the cell as a whole being measured for each successive withdrawal of the plates, the sum of the calculated conductances of these prisms, between the electrodes, at the side of the electrodes, and directly under the electrodes, was subtracted from the measured conductance of the whole cell in order to

determine the residual conductance ascribable to the bottom diagonal prism. The number of factors to be considered in this measurement left the determination of the conductance of this bottom diagonal prism rather unsatisfactory. From three or four carefully conducted experiments, however, we can reasonably conclude that the conductance of a relatively large diagonal prism, such as would be encountered in practice, does not appear to add sensibly to the conductance of the cell as a whole.

Practical Conclusions.

There results from this study the conclusion that the practical conductance of the electrolyte situated between the two electrodes (including that directly, laterally and beneath the electrodes) can be calculated by adding to the conductance of the portion between the plates the virtual conductances of the side and bottom prisms. These latter are to be determined in each case by calculating from the respective total conductances of the prisms (as if between parallel plates) and the use of the percentage conductance as given in the curve in Fig. 2. It is necessary to reckon the ratio of depth (distance from edge of plate to side or bottom of tank) to length (distance between the plates), refer to the curve and find for this ratio the percentage of the total conductance of each prism actually available. The conductance of the diagonal bottom prism may be neglected in all cases in actual practice.

For practical purposes, where the ratio of depth to length happens to be greater than 0.8, the conductance of the prism of electrolyte beneath or alongside of the electrodes may be considered as constant, and calculated as equal to the total conductance (between parallel end plates) of a prism of electrolyte of the length concerned and one-quarter as deep as it is long. Or, in other words, the maximum conductance of these lateral or bottom prisms of electrolyte is reached when their depth (measured away from the plates) is over 0.8 of their length (distance between the plates), and may be calculated as the total conductance of a prism of solution whose depth is one-quarter of its length.

There are various other more complex problems in this connection, particularly in tanks used in electro-plating where the plates are not against the ends of the tank, or between uneven

surfaced electrodes, but the infinite variety of such cases makes a satisfactory study of them impossible. We have worked out the simplest case such as we think will assist the electrochemical engineer in the design of ordinary metal refining tanks.

The fact that there is reached a maximum conductance after a given depth, holding for both electrolytes and metals, is important as showing the uselessness of increasing the depth of the electrolyte underneath or at the sides of the electrodes past a certain point, such increase in dimensions adding nothing to the conductance. The uselessness of increasing the depth (width) of an electrical metallic conductor conducting between points of contact at the two ends on the same side, beyond a certain depth which is approximately the ratio of depth to length of 0.8, is quite as important for the electrical engineer in the design of equalizing bars and heavy bus bars of short lengths, and shows the great importance of making contact over the whole end or face of the conductor in order to get the full utilization of the metal contained in it. Connection should never be made simply along an edge or at the corners.

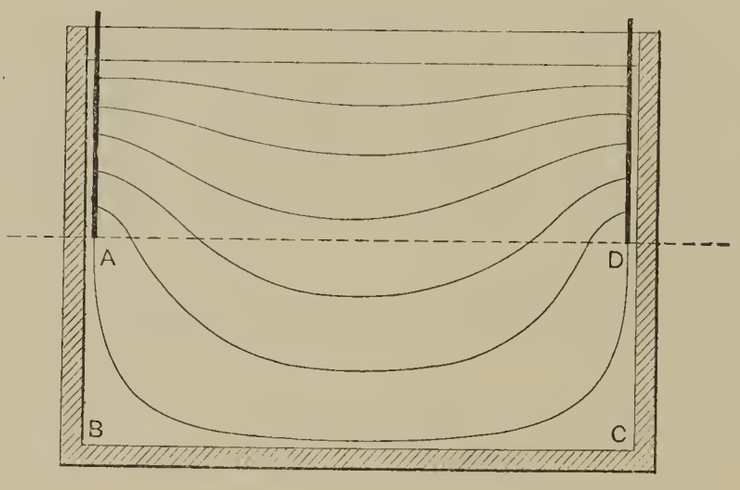
Metallurgical Laboratory, Lehigh University.

DISCUSSION.

DR. W. R. WHITNEY: I want to discuss this paper for a moment. It is a very interesting question which Prof. Richards has brought up. I recall the first time I learned the general fact, and I only rise to put it in another way, so that we will all have it as part of our electrochemical equipment. You will find by experiment that when you use relatively small electrodes, as, for example, by putting wires into a vessel containing an electrolyte, the distance between the electrodes has very little to do with the actual resistance. One would at first thought expect the resistance between such electrodes to be almost proportional to the distance between them. When one employs conditions in which the electrodes are relatively large, the resistance begins to be more nearly proportional to the distance between them, and in cases where the electrodes fill the cross-

sections of the electrolyte, then the distance between the electrodes determines exactly the resistance of the solution.

Mr. Townsend Wolcott: I am surprised that nobody has anticipated the result which Dr. Richards has brought forth. He considers that the current in the lower portion of the electrolyte, A, B, C, D, emanates from the lower points of the electrodes, A and D. As a matter of fact, the lines of flow go down into the lower portion of the electrolyte from all points of the electrodes; strictly speaking, there are no perfectly straight lines of flow. After adding a certain depth of electrolyte, one would not expect to increase the conductivity much by still further additions to the depth. An analogous case is the flow between



vertical cylindrical electrodes through the electrolyte. This is a well-known case of lines of flow and equipotential surfaces, and it is stated in all text-books that, after they are moved 10 or 12 diameters apart, it makes very little difference how far apart the electrodes are, supposing there is unlimited electrolyte all around. The whole matter is simply a question of the mathematics of the lines of flow. When the electrodes are 10 or 12 diameters apart, the cross-section of the middle portion of the path of the current is increased so much that the resistance of this portion is small compared with that in the vicinity of the electrodes, and it makes very little difference if they are moved still further apart. The experiments agree with the accepted theory, provided there is no complication from polarization or anything of that kind; then it is simply a matter of the flow of current according to the laws of metallic conduction.

DR. MORRIS LOEB: If I understand the previous speaker correctly, I agree with him that you are dealing with a phenomenon not very dissimilar from that of radiation of heat from different points of a metallic bar whose opposite ends are at different temperatures. It would be a great mistake, in such a study, to consider the variation of conductance as due simply to distance between the electrodes, or, in fact, consider the electrodes themselves as determining points. You might exclude the electrodes from consideration, and, if in any two sections of a liquid column there were differences of potential, conductance phenomena must necessarily appear. If Professor Richards were to work this problem out mathematically, I do not doubt that he would find the law not very different from Fournier's equation for thermal conductance as applied to diffusion phenomena.

MR. ELMER A. SPERRY: I wish to add my word of commendation on this paper. It certainly has a very practical bearing. I have some curves, determined years ago from the resistances of water rheostats. You understand, abroad water or liquid rheostats have met with considerable use in traction work, and curves were prepared of the resistance with one, and also both of the electrodes immersed to varying depths. Starting with the curves I had of that type, we determined how much the apparent conductivity of the electrolyte was affected when one electrode was lessened in size. As against the infinite area of one electrode, if you diminish the other, the effective conductive area is double what I understood the author to say he found when both electrodes are raised; that is, you find a maximum conductivity, which, instead of being 50 per cent., is an expression equaling 100 per cent. of the distance between them, and beyond that it does not seem to have very much effect. The practical value of this whole question is well illustrated where graphite or some similar expensive substance is used as one of the electrodes; the question is how much can you diminish the electrodes without diminishing efficiencies.

MR. CARL HERING: Several years ago I made a statement before this Society in regard to an old experiment which, I believe, Lord Kelvin made many years ago. He took two plates, each a foot square, placed them in the ocean at various distances

apart, and measured the resistance between them. He found that this resistance, after they were a certain distance apart, was practically the same for all distances, which, as Mr. Wolcott has just explained, is also shown to be the case by theoretical considerations. After the distance apart was relatively large as compared with the size of the plates, the resistance became a function of the size of the plates rather than of their distance apart.

Another remark made at a previous meeting was to the effect that the resistance of the ground was measured by someone between various points, and it was found that, beyond a certain distance, it did not make any difference how far apart the ground plates were placed, the resistance was always the same.

PRESIDENT BAEKELAND: In connection with this subject I would have liked to hear Mr. Sperry call your attention to a matter directly connected with the practical conductance of electrolytes, namely, the influence on practical conditions brought about by the gas bubbles; this depends on the strength of the current. I remember a case where all the calculations were totally upset in practice through this cause. It was not a case of polarization, but the mechanical interposition of moving gas bubbles. We found that there was a limit, in fact, to the useful dimensions of the electrodes in one direction on account of that particular phenomenon.

PROF. J. W. RICHARDS: Mr. Wolcott remarked that if no polarization occurs at the electrodes, these results are true. We avoided polarization at the electrodes, and the results were exactly the same as if it were plain conduction. The conduction was in one case in an electrolyte, and in others experiments in the metals—true metal conduction—yet the two curves practically overlapped each other. I think this may be regarded as another proof of the position, which I have taken for several years, that as far as conduction in electrolytes is concerned, considering the electrolyte merely as a conductor, there is an identity between the conductivity of an electrolyte and metallic conduction; you can make no distinction in kind between the conduction of electricity through the body of an electrolyte and the conduction of electricity through any other conductor.

MR. HERING: In reply to what the Chairman has said, I might cite as an interesting illustration that processes were described some years ago in which the resistance at the electrode was made so high that the electrode became red hot and even melted while under the liquid. I had the pleasure of seeing this experiment repeatedly; it was, in fact, proposed at the time to use this method of heating iron in place of the blacksmith's forge, for when the iron was made the cathode there was never any oxidation or burning of the metal.

DR. RICHARDS: I believe that electrochemists having to calculate or design electric metal refining baths will understand well the bearing of our tests upon the resistance of the bath. Lateral, upper or lower prisms of electrolyte adjacent to the plates have a maximum of auxiliary conductance when their depth equals the distance between the plates, and this maximum is equivalent to the calculated conductance of a depth of solution equal to one-half the distance between the plates. The use of these determinations will help to make more precise the calculation of the resistance of a refining bath.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909; President L. H. Baekeland in the Chair.

CERTAIN CHARACTERISTICS OF DRY CELLS.

By Charles F. Burgess and Carl Hambuechen.

A dry cell, like any other electrical apparatus, must possess certain characteristics to enable it to perform the service demanded of it. It is impossible to determine or define in simple terms the desirable characteristics of a dry cell, since this depends upon the service with which it is to be associated.

The greatest usefulness of the dry cell at the present time is probably in the telephone field. The service next in importance is ignition as effected through spark coils; electric bell and annunciator work furnishes the next largest field for dry cells, and the operation of telegraph and alarm circuits, electric clocks, phonographs and other small electrically driven machinery are some of the other uses in which this electrical generator is extensively employed. New uses are continually being evolved, and the demand for dry cells appears to be continually increasing, and this in spite of the fact that the central energy systems for telephone exchanges are displacing large numbers of dry cells, and magneto and other forms of electrical ignition are competing with the dry cell for the ignition business.

A great majority of the uses of dry cells involve relatively low rates of delivery of energy. The cells are called upon intermittently, and the primary requisite of a dry cell appears to be its ability to live over a long period of time. The attainment of this characteristic is shown in marked degree in certain cells on the market, and in this respect marked improvement has been effected during recent years.

Low internal resistance is another important qualification for a satisfactory dry cell, since this enables the cell to deliver its energy to the external circuit with high efficiency. Where very small quantities of energy are required, at a low voltage, low resistance may not be requisite; in other uses where the

instantaneous demands upon the battery may be great, low resistance is a most important characteristic. Low internal resistance is usually indicated by high amperage, *i. e.*, the ability of a cell to deliver a large amount of current when short-circuited through an ammeter.

"Minimum polarization" and "rapid recovery" are other factors to which attention is called by manufacturers in advertising their dry cells.

It is impossible to point out which is the most important of these qualities, since the relative importance depends upon the character of service required. Among the minor characteristics which are sought to be attained in dry cell construction are: Absence of the leakage of the liquid, minimum increase of resistance with low temperature, convenience for connecting into circuit, and the general appearance of the cell.

The factors which govern the above characteristics include these: Materials employed in construction, the proportions of materials, the chemical and physical qualities of these materials, methods of assembling.

In spite of the fact that 40,000,000 or thereabout represents the number of dry cells manufactured each year in this country, little scientific work has been done in the way of making a detailed study of the various individual factors which influence the quality of the cell, if we may judge by the meager publications. On the other hand, much scientific work has undoubtedly been done, if we may judge by the marked improvements which have been effected in the product. It appears that dry cell manufacturers who are doing the larger part of this work do not feel inclined to discuss analytically dry cell construction.

Perhaps 80 per cent. of the dry cells made and used are of the so-called No. 6 size, being in cylindrical form, the zinc container 6 inches high and 2½ inches in diameter. As far as the cylindrical zinc container is concerned, there is striking uniformity in dimensions and costruction, the principal difference being in the thickness of the zinc, which may be found to vary from No. 7 to No. 11 sheet zinc gauge.

All cells employ a carbon rod for the cathode plate. In some forms of cells this rests on the bottom layer of paper, and in others it is raised a half inch from the bottom. It extends up

through a pitch seal, and a brass cap or brass screw attached to the top constitutes the positive terminal of the cell. Some of the carbons used are of smooth cylindrical shape, while others are corrugated or fluted to give greater contact surface to the filling material.

Another striking similarity in most of the dry cells is the list of materials which are introduced into the zinc container. Similarity then ceases, and we encounter variations in the quality of the materials, the proportions, and in the methods in which the materials are introduced into the container.

The writers are unable to refer to published formulæ which are descriptive of the better type of cells at present in use. The following list and proportion of materials may be taken as fairly representing the filling mixture in well-known types of cells:

10 lbs. of manganese dioxide.
10 lbs. of carbon or graphite, or both.
2 lbs. of sal ammoniac.
1 lb. of zinc chloride.

Sufficient water is added to give proper amount of electrolyte to the cell, and the most suitable quantity depends upon the dryness of original materials, fineness, quality of paper lining, etc.

While these are the essential elements, other materials are frequently added—such as starch or other forms of paste, to improve contact of electrolyte to zinc and promote distribution of action throughout the charge. Mercury is occasionally added to effect amalgamations and prolong life of the zinc.

With the materials as above listed and in the proportions named the resulting cells may be good or they may be practically worthless, depending upon various factors. It is generally believed that purity of materials is the pre-eminent requisite. Commercial conditions, however, prohibit the use of chemically pure materials. The customary specifications for manganese dioxide call for a granulated or powdered material having about 85 per cent. of MnO_2 and less than 1 per cent. of iron. This is a specification which can readily be complied with, and by careful selection and concentration it is possible to get as high as 92 per cent.

Physical qualities, porosity and size are grains are likewise important. The amount of available MnO_2 differs according to the chemical test employed, and "available MnO_2" by test may not be the available MnO_2 under the conditions existing in the cell. In storage batteries PbO_2 is an active material only when in suitable contact or proximity to grid or framework of the positive plate; and MnO_2 in a dry cell is active to a degree, depending upon its contact with the conductive carbon constituting the negative electrode. It is evident that penetration into the interior of a piece of MnO_2 is necessary if the entire mass is to be active; and porosity seems therefore to be important.

It is perhaps superfluous to point out that the purpose of the manganese is to effect depolarization, and thereby to maintain the voltage of the cell during discharge. The qualities designated "minimum polarization" and "great recuperative power" are dependent largely upon the amount and availability of the MnO_2 employed.

Carbon in a powdered or granulated form is employed to give conductivity to the mass of MnO_2 and to furnish a large amount of cathode surface. Retort carbon, coke, petroleum coke and ground carbon rods and electrodes are the various grades of carbon commonly employed for this purpose, and much of the variation found in dry cells is due to varying qualities of this carbon. Graphite is another form of carbon extensively employed in dry cells, and the more recent improvement in dry cells is undoubtedly due largely to the liberal use of this highly conductive though more costly form of carbon.

Sal ammoniac of a very pure grade is generally employed, and in addition to purity, the physical qualities of dryness and freedom from lumps are requisite to ensure proper mixing with the carbon and manganese.

The zinc chloride is also required to be of high purity and to be free from iron.

Sheet zinc constitutes the anode and serves at the same time as a container. Purity is naturally of importance. The purchaser does not have, however, a variety of grades of zinc to choose from.

This anode makes contact with the electrolyte in the form of paper saturated with solutions of zinc and ammonium chlorides.

Ideal conditions call for a corrosion of zinc only in amount equivalent to the current delivered to the external current. Excess of corrosion above this amount is caused by local action, and one of the principal problems in dry cell manufacture is to reduce such local action to a minimum.

There are numerous possible causes of local action. A particle of metal other than zinc, making contact with the zinc electrode surface produces a galvanic couple. Since solder is employed on the side seams and bottoms, some of which frequently finds its way inside the cell, it might be natural to assume this as a most frequent cause of local action. This appears, however, not to be the cause, for while solder is decidedly electronegative to the zinc, it is composed of two metals, lead and tin, both of which have high "over-voltages" which almost entirely prevent current flowing to these metals at the expense of and under the electromotive force set up by the zinc.

Some experimental cells made from sheet zinc streaked with solder on the inner surface showed little if any abnormal deterioration due to local action even after standing for several months.

On the other hand, if the couple be made of copper, or iron in contact with the zinc, the "over-voltage" is not sufficient to prevent the flow of current, and destructive local action occurs.

Copper occasionally finds its way into the dry cell from the brass terminals through corrosion and the diffusion of the copper chloride into the cell. Upon reaching the zinc the copper is deposited and establishes a couple. A small amount of copper placed in a freshly made cell may cause it to run down completely in less than one day, the entire zinc being corroded much as though the cell had been used to its limit.

Iron is less harmful than is copper, and it is something of a question as to just how far it is economical to go in excluding iron from the cell. This seems to be the principal substance which specification of materials seems to exclude. The manganese oxide and carbon are considered the more valuable the lower the percentage of iron contained. This iron cannot be excluded, being present to the extent of perhaps 1 per cent. But this iron cannot be harmful unless it goes into solution, though

diffusing to the zinc. The electrolyte is such that dissolving of iron is improbable, since the liberation of ammonia in zinc and ammonium chloride solutions tends to throw out iron.

It occasionally happens that some of the filling material, the manganese oxide and carbon, gets between the paper lining and the zinc. This naturally gives rise to damaging galvanic couples. Each of such particles produces a zone in which corrosion of zinc takes place, and if such contact of carbon and manganese oxide be perforated the effect on the cell is the same as though it were short-circuited.

Inequalities in the zinc itself may cause certain differences in potential, and therefore give rise to galvanic couples. The inequalities of temperature due to soldering give rise to small differences; the solder applied to the surface penetrates into the metal and possibly alters the contact potential in the other side. What appears to be a most frequent cause of local action, and most difficult of control, is the access of air to the active surface.

Some measurements on these potentials have been made by placing between two pieces of sheet zinc a paper soaked in battery electrolyte, the paper having been previously painted around the edges to exclude the influence of the air. The pieces of zinc were then pressed tightly together and differences of potential measured on a potentiometer. Two pieces of zinc cut from different portions of a sheet give slight differences of potential, the maximum value among a number of tests being 0.002 volt, and usually less. Upon heating and slowly cooling one of the pieces of zinc, to duplicate the influence of the soldering temperature, potential differences varying between plus and minus 0.012 volt were observed. Heating and rapidly cooling by quenching had a much smaller influence on the potential. Solder on the reverse side of one of the test electrodes had an influence amounting to 0.004 to 0.007 volt. The influence of air getting to the electrode surface was tested by first perforating one of the test electrodes and pressing it against another piece of unperforated metal with the saturated paper interposed. The first measurement showed a potential of 0.0026 volt, which gradually increased until after two days it had risen to 0.0224, the perforated plate being plus. This is a potential difference

sufficiently high to cause somewhat rapid cell deterioration, and points to desirability of excluding as far as possible the access of air to the interior of the cell.

If we were to assume all inequalities of the zinc electrodes to be removed, we still have opportunities for local action due to inequalities in the electrode on the other side of the soaked paper. Non-uniformity in the mixture is detrimental. Some manufacturers place the carbon rod in direct contact with the saturated paper resting on the bottom of the cell, the mixture surrounding it. This must necessarily give rise to some difference of potential, and a measurement of this gave a value of 0.176 volt, in such direction that current tends to flow from the carbon through paper to zinc, thence through the paper to the manganese mixture and back to the carbon. To prevent this source of local action is probably the reason for the majority of cells now being made with the mixture interposed between all portions of the carbon surface and the paper.

It is evident, when we consider the numerous possible sources of trouble leading to faulty cells, that the making of a good dry cell requires care. Amalgamation of the zinc is occasionally resorted to, but it is doubtful whether material advantage accrues through the use of mercury in the dry cell.

It is customary to consider the carbon rod as the cathodic electrode, and in this we have arguments favoring the large corrugated and fluted shapes over the smaller cylindrical shapes, in that greater active electrode surface is thereby attained. It is doubtful, however, whether the surface of this rod is to an appreciable extent an active electrode surface. It makes contact with and is surrounded by a mass of material—carbon, graphite and manganese—possessing metallic conductivity even though moistened by electrolyte. It would therefore be more accurate to consider the active cathode surface as that portion of the mixture just within the paper. While this may be the main cathode surface, it is not exclusively so, and the active surface progresses inward as the action of the cell goes on.

The cathode is a compound one, consisting of an intimate mixture of conductive carbon and graphite in contact with manganese oxide, and the electromotive force of the cell is the summation of the electrode potentials, 0.56 volt for the zinc and

about 1 volt for the carbon-manganese oxide mixture, or a combined voltage of 1.56.

While the cell delivers current to the external circuit this voltage decreases, the rapidity depending upon the amount of current drawn. If it were not for the manganese dioxide the drop in the voltage would be very rapid, and the value of the cell is therefore dependent largely upon the active manganese oxide or depolarizer. This manganese oxide is consumed. As commonly stated, it is reduced to Mn_2O_3, and in undergoing this change it becomes inactive both as a depolarizer and as a conductor. It is probable that the MnO_2 possesses sufficient metallic conductivity to contribute something to the conductivity of the cell, and when changed to the lower oxide it loses this conductivity. It thus becomes inert and occupies valuable space. It is natural to assume that the MnO_2 in the outer portions near the paper is the first to become reduced, the action progressing inward to a certain depth, this depending upon various factors, such as the intimacy of contact between the carbon and MnO_2, the purity, fineness and porosity of the latter, etc.

If the MnO_2 possesses metallic conductivity and makes contact with conductive carbon and both are moistened with electrolyte, there is a possibility that a certain amount of chemical action occurs owing to the voltaic couple thus produced.

A study of what actually takes place, chemically and electrically, inside of the dry cell while in action, and while standing in storage, offers an excellent subject for analytical research. While the chemical reactions which take place in the dry cell or other cells of the Leclanché type are frequently explained in a simple manner in text-books, a careful study will show that simplicity does not exist.

The electrical energy output of a dry cell is equal to the ampere-hours multiplied by the voltage. The current which a typical cell will deliver when short-circuited and "run down" in one hour is in the neighborhood of ten ampere-hours. It may go as high as thirty ampere-hours or more as the rate at which the current is drawn is diminished.

The open-circuit voltage is almost universally 1.5 to 1.6 volts.

The useful or effective voltage is less than 1.5 volts, and an average of 1 volt is a fair figure to assume at the cell terminals

during a discharge at a rate such that thirty ampere-hours may be drawn. With an electromotive force of 1 volt and a current output of thirty ampere-hours, a fair energy rating for a dry cell is thirty watt-hours.

The voltage at the cell terminals while delivering current naturally depends upon a number of factors, the principal of which are the activity of the depolarizing material and the internal resistance of the cell. The activity of the depolarizer increases as the cell is used, and even more markd is the increase in resistance with the number of ampere-hours delivered.

It is very commonly believed that the increase in resistance is due to the drying out of the cell, but the drying is of very minor importance as compared with other factors. One of these is the increase in resistance due to the partial reduction of the MnO_2. Another is the accumulation, on the surface between the paper and the zinc, of non-soluble impurities of the zinc and the various basic salts and double salts resulting from the electrolytic corrosion.

When a cell is first made up the paper is saturated with a solution of zinc and ammonium chlorides. More zinc chloride is added as a result of the corrosion of the zinc. The increasing concentration of the $ZnCl_2$ in the presence of the ammonium chloride gives just the conditions which are needed for the production of various double chlorides, some of which are much less soluble than the single chlorides.

These insoluble double chlorides occupy the pores and spaces in the paper, crowding out the electrolyte and increasing the resistance. If ammonia is liberated, it may react with the $ZnCl_2$ to form a precipitate of ZnOH, which likewise clogs the cell, unless suffiicent ammonia be present to re-dissolve this precipitate. That this clogging action occurs at the paper and proceeds inward toward the carbon rod is evident, since in taking apart a run-down cell there is usually found a hard crust of an eighth of an inch or more in thickness just inside the paper.

It is no doubt true that improvement in dry cell construction must come through reducing this accumulation of insoluble non-conductive materials. It may be suggested that this precipitation due to double chlorides might be delayed by using only NH_4Cl in the electrolyte to begin with, and, in fact, a somewhat

greater ampere-hour output can be obtained in this way. But it involves the more serious disadvantage of shortening the life of the cell on open circuit. The presence of $ZnCl_2$ in the electrolyte decreases in marked degree the local action.

There is no doubt that marked improvement has been made in dry cells during the past few years, and there is no reason to believe that perfection has yet been approached. The field for research in the study of this important device has by no means been fully occupied.

From the technical point of view the need for and possibility of improvement are equally great. The common and grossly inadequate methods of rating dry cells in terms of the number of miles they will run the ignition device on automobiles, or the number of months they will stand up on telephone service, or the number of sparks that they will produce, all of which are dependent upon the kind of device and the nature of the same in connection with which they are used, should give way to rating by energy output.

This can only be done after determining and generally adopting the standard methods for conducting tests.

Chemical Engineering Laboratories,
University of Wisconsin.

DISCUSSION.

MR. CARL HERING: I have been very much interested in this paper, and I am glad to see that we are at last getting some able contributions on this subject.

The paper states that the electromotive force of the cell is the sum of the two electrode potentials, 0.56 volt for the zinc and about minus one volt for the carbon-manganese oxide mixture, giving the combined voltage at 1.56. It seems to me that there must be something wrong in this. It credits the greater part of the energy to the reduction of the manganese oxide, whereas it seems to me the energy comes chiefly from the oxidation of the zinc. I would like to ask, therefore, how it was determined; if

it was determined by measurement, was not the method faulty? It seems that no one has yet been able to measure the absolute single potential of an electrode by a method which is really beyond question. If Prof. Burgess has found a method I would be very glad to know what it is. Calculation would, I think, show that the zinc gives the greater part and the manganese oxide the lesser part of the energy. Generally, the energy contributed by the depolarizer is very small.

The paper also says it would seem better to reduce the formation of the insoluble compounds, that is, that it would seem better to have them soluble. The question arises whether that is really desirable. There is only a very small amount of water in the battery, and that small amount has to do a great deal of work; it seems to me that if the products were soluble rather than insoluble, the water would be used up more rapidly, by having to hold in suspension the products that are formed; it would, therefore, not be available any longer as water. If this is correct, it seems to me that it is rather a desirable thing to arrange to have the chemical products formed insoluble, so that they will deposit out and leave the water there to keep the cell wet. The latter reminds me of an old definition, namely that the dry cell is so called because it always remains wet, in order to distinguish it from a wet cell which may run dry.

Mr. Charles L. Parsons: I would ask Prof. Burgess if in his experiments with dry cells he made any experiments in the use of bromates as depolarizing and oxidizing agents. In some work we have done during the last year bromates certainly showed wonderful oxidizing power at low temperature. I am interested to know whether these bromates have been worked with to any extent in dry cell construction. They are getting so that they can be obtained quite cheaply.

Mr. J. A. Seede: Will Prof. Burgess tell us, approximately, the purity of the zinc in each cell and the weight of the zinc in each cell, also what does he mean by the statement towards the bottom of page 100 that the purchaser does not have a variety of grades of zinc to choose from.

Prof. Burgess: At the bottom of page 103, where the potential of the zinc is given as 0.56, it is an oversight not to state that that

is based upon the use of the so-called normal electrode. If Mr. Hering will look up the Ostwald series of potentials, for the potentials of metals, he will see that that is the value given for zinc. It is simply an arbitrary designation, based on a somewhat arbitrarily chosen zero point.

As to the use of water, this is needed between the zinc and mixture, and if the space which should be occupied by that water is taken up by insoluble materials, crowding the water further back into the mass, the electrolyte is not where it is needed.

As to the bromates, chlorates and many other oxidizing compounds, the use of these in most instances will increase the voltage of the cell and increase the amount of current output, but in general my experience has been that the soluble oxidizing agents are detrimental to the life of the cell, that is, they increase the local action.

My statement about the purity of the zinc is based upon statements which have been made to me by dealers. There appears to be practically one grade of zinc obtainable, it is only a high quality of zinc which can be satisfactorily rolled. The users do not appear to be able to dictate how pure a grade of zinc shall be furnished to them, and they take the best that can be obtained. An analysis of the sheet zinc can, of course, be made—it is some time since I paid any attention to it, and I cannot give the figures for the purity of the zinc which is being used.

Mr. S. P. Sharples: There are many grades of zinc on the market, and we have been in the habit for years of testing zinc for the ordinary wet batteries. The requirements are now that it shall contain only traces of lead and no iron, and such zinc is readily obtainable on the market. I will mention one brand on the market which we have used, and that is zinc from the Bertha mine, in Virginia, and it is well adapted to use in batteries.

A paper read at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909. President L. H. Baekeland in the Chair.

DRY CELL TESTS.

By F. H. Loveridge.

If we are to judge by numerous inquiries from users of "dry cells" as to the electrical qualities which such cells possess, the conclusion is reached that ideas along this line are very indefinite. Many users consider that a cell is good for a certain length of time, and that this period is not affected by the rate at which energy is used nor the way a cell is treated.

Among causes for this impression might be mentioned the delay that sometimes occurs between the making of cells and their going into service—dealers as well as users may hold cells in stock for excessive periods and under conditions that are unfavorable. Faulty circuits may permit heavy current drain and proportional shortening of useful life. A cell may become apparently exhausted and later recuperate to a point where it again becomes useful. Such causes are conducive to a belief in erratic and irregular action.

Efforts along various lines are sometimes made to get definite information as to what may be expected of a cell, but one of the great obstacles encountered is the fact that any electrical test that may be made which consists of instrument readings occupying a few minutes or even hours is useless in predicting what a cell will do under various conditions of service, unless, of course, the make and characteristics of the cell are so well known that readings simply serve the purpose of fault detectors.

It is natural then, under conditions which do not readily give exact results, to find opinions based on impressions rather than data, but this does not alter the fact that the properly designed and constructed dry cell of today when properly used is a very dependable article, and its output of electrical energy for any given class of service may be very accurately determined.

It would seem to be a very desirable thing to have dry cells rated on an ampere-hour or watt-hour basis under some definite

condition of energy discharge, but such a thing is practically unknown at the present time. The rating we now have is that of dimensions of the cell and its current. The first might be accepted as reasonable, but the second is misleading. Amperage ratings are high and apparently based on the instantaneous current a cell gives when short-circuited through a low resistance ammeter; such readings are of practical value in discovering defects, but so far as determining an energy output is concerned, they are of little value because the sustained current a cell can give has very remote relation to the short circuit current. The voltage reading is also useful in discovering defects, but inasmuch as the cell may give out a very considerable part of its energy and cause only a small drop in this value it follows that such readings are not of much importance.

The "shelf test," or, in other words, the test made after a cell has remained for a certain time in storage, is one that serves a useful purpose for the reason that it is desirable to know the change that takes place during such period. Very few cells go into service immediately after they are made, and during the delay more or less deterioration takes place.

There is, however, the difficulty that if the test comprises readings of current with a low resistance ammeter, a decrease of 50% in its value would indicate only an insignificant increase in internal resistance, which would be unimportant with ordinary current drains. If current readings are taken with a resistance inserted in series, then the variation showing the change in internal resistance is correspondingly reduced.

Where it is desired to take the current and voltage of a cell, it should be done by means of instruments that do not absorb a large part of the energy in themselves. Hence a high resistance voltmeter and a low resistance ammeter should be used—a minimum of 150 ohms for the voltmeter and a maximum of 0.01 ohm for the ammeter are found to be satisfactory values.

Taking everything into consideration, we are finally brought to the conclusion that the best shelf test is one that compares the actual performance under working conditions of the cell after a certain length of time on shelf, with that of a new cell. In this way results are obtained that involve no factors of uncertainty. There is involved in this the using up of cells to find out what

they will do on life test. Such procedure, however, seems necessary since other methods involving tests not absorbing energy are not dependable and sample cells used up on life tests do not represent very great expense.

Deterioration is shown by an increase of internal resistance, inasmuch as voltage readings decrease very slightly on shelf. It is impossible to give any exact data as to the increase of internal resistance on the shelf, because there is such a wide variation even in cells of the same make, but, roughly, it might be said that a reduction in the short circuit current of 50% would be a fair average in one year at ordinary temperatures.

The question of change in internal resistance from change in temperature is sometimes brought up. I recall one test in which

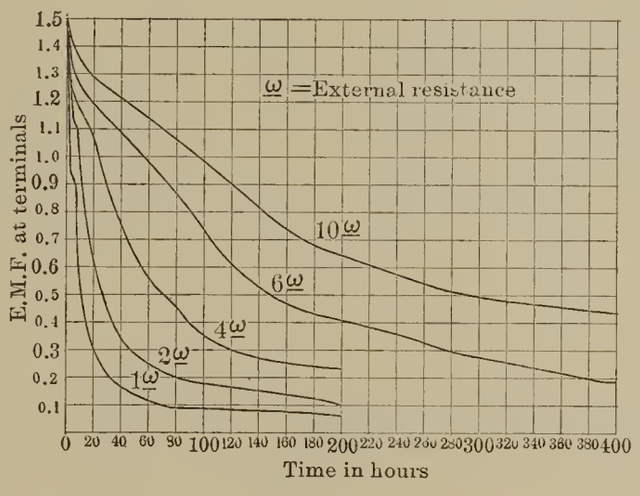


FIG. 1.

a decrease from 100° F. to 25° below zero F. produced an increase in internal resistance of only about 100%—even at this latter temperature the internal resistance was so low as not to interfere seriously with a light current drain. The indications in this test were that the increase of internal resistance was proportional to the decrease in temperature.

Life tests are often made on dry cells by connecting them permanently in circuit with a fixed resistance. This method is the easiest one to employ, as it requires no attention other than the taking of readings at the desired intervals. Such a test is of no great value, however, as the energy output is small and the life very short unless the current is kept low. Under these conditions the staying qualities of the cell are not demonstrated, and the ability of the cell to stand up under ordinary current drain after

a considerable period on "open circuit" service cannot be predicted with safety.

In Fig. 1 is shown the result of tests made on dry cells of the standard 2½" x 6" size, in which the circuit is closed continuously through a fixed resistance, the resistance in each case is indicated by the figure associated with the curve. The ordinates give the potential difference at the terminals of the cell and the abscissas the time in hours. It will be seen from an inspection of these curves that there is a very rapid drop in potential at the terminals of the cell when the resistance is small. As the resistance increases the length of time required to reach any given potential difference is increased. Thus after ten hours the potential difference at the terminals of the cell has reached 0.5 volt where the resistance in circuit is 1 ohm, whereas with 10 ohms in circuit a value of 0.5 volt is reached after 285 hours.

There is a wide variation in the energy derived from cells under these varying conditions as would, of course, be inferred from the forms of the curves. The value of this energy output in the different cases has been figured to a point at which the potential difference has fallen to 0.5 volt and is found to be as follows:

With	1	ohm	7	watt	hours
"	2	ohms	8	"	"
"	4	"	14	"	"
"	6	"	21	"	"
"	10	"	24	"	"

These values would, of course, be modified if the integration were carried to lower points with the lower resistance, but inasmuch as 0.5 volt is only one-third the initial electromotive force, it is seen that we are carrying our energy drain to quite a low point and that the volume of current has fallen to one-third its original value. These curves show approximately what may be expected as the characteristic performance of dry cells under the above conditions.

As we have seen that the "closed circuit" test is not a safe one for "open circuit" work, the best test we can apply is the "open circuit" one in which the cell may be made to show what its life characteristics are when its energy is used over a relatively long period. Sample cells are generally taken for this purpose and tests should duplicate working conditions as nearly

as may be. Some form of clock controlled contacts are very well adapted to this purpose and may be made to control the open and closed circuit periods in any manner desired. It is also a good plan to use relays controlled by clock contacts for closing the test circuits. By this means, the master or pilot contacts on the clock are only used for light currents, and it is much easier to maintain them in good condition. The mechanism and contacts should be well made and reliable, as it is difficult at best to get consistent results. The shorter tests as a rule give results more satisfactory as regards uniformity than long ones—the chances for variable factors entering being greater with increased time.

In taking readings on dry cells, it is, of course, necessary in either "closed" or "open circuit" tests to have some form of voltmeter to measure the voltage. It is also desirable to use such an instrument for current readings employing the fall of potential method, as this enables current readings to be taken without disturbing circuit connections. If an ammeter is inserted in circuit from time to time, there is a tendency to vary contacts and resistance. Anything tending to introduce variations should be carefully avoided.

In Fig. 2 is shown a set of three curves representing the relation between electromotive force (curve A), potential difference at the beginning (curve B), and potential difference at the end (curve C) of the closed circuit periods taken from an open circuit test of a standard dry cell.

The electro-motive force is seen to be a gradually diminishing quantity, which, however, remains at a comparatively high level. At the instant of circuit closure the potential difference immediately falls and the divergence of curve B from curve A indicates the increase in internal resistance. Curve C also diverges from curve B, thus indicating polarization during the closed circuit period. The lowest curve is in reality the one of greatest value, for it indicates the level at which the cell maintains its potential under service conditions. These curves are also of interest as showing what may be expected in the way of internal resistance.

The question is often asked as to the magnitude of the internal resistance of dry cells. From the above curves it is seen that there is a continual increase in this quantity as energy is given out. We know also that the internal resistance is very low

when the cell is new, for the current on short circuit is high; as the cell grows older on the shelf the resistance is continually increasing, though to a much smaller extent than when it is on even very light service. In attempting to answer the question, it is necessary to take both these factors into consideration. Hence the requirement for a definite answer is a definite statement of conditions.

In Fig. 3 four curves are shown which are plotted from the results of intermittent tests, each curve representing a special condition. The values are taken from the potential difference at the end of closed circuit periods. As will be seen from the data, curves A and B represent the same external resistance, which is about 1.6 ohms. Curves C and D represent the same conditions,

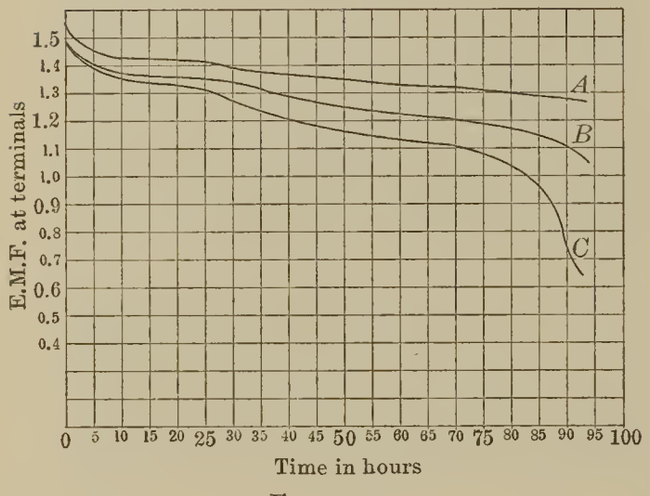


Fig. 2.

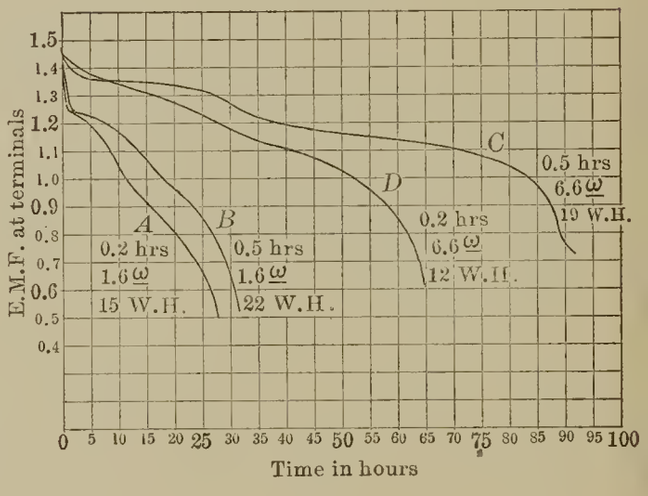


Fig. 3.

but with 6.6 ohms. Curves B and C represent conditions where circuit closure averages about 0.5 hours per day and curves A and D represent an average of about 0.2 hours per day.

An integration has been made to an arbitrary point where the potential difference has fallen to 0.7 volt. Where the resistance in circuit is low the watt hours are higher than with corresponding time conditions and high resistance. It seems from this that, down to the integration point taken, cells will give out more energy when allowed to do so at the high rate. At the low rate the energy, though less in watt hours, extends over a longer period of time and the potential difference is maintained at a much higher level. In the tests in which the same resistances are used but the time varied, it will be noted that the watt hours are less with the smaller daily period. This is to be accounted for largely

by deterioration taking place over a longer period of time. It is probable that it is due in large measure to the fact that when current is drawn from the cell the action is not uniform,—this lack of uniformity would tend to set up potential differences in the cell itself. The result would, of course, be the setting up of local action which would deteriorate the cell to a degree determined by the unbalancing. A degree of unbalancing is to be expected which bears some proportion to the magnitude of current. The energy of curve A is less than of curve B by about the same amount that curve D is less than curve C, but C and D represent much longer periods of test, hence a smaller rate of deterioration.

In Fig. 4 are two curves which represent tests similar to that

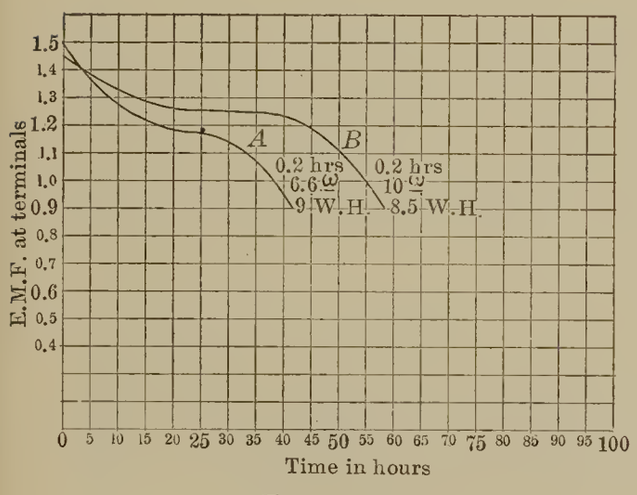


Fig. 4.

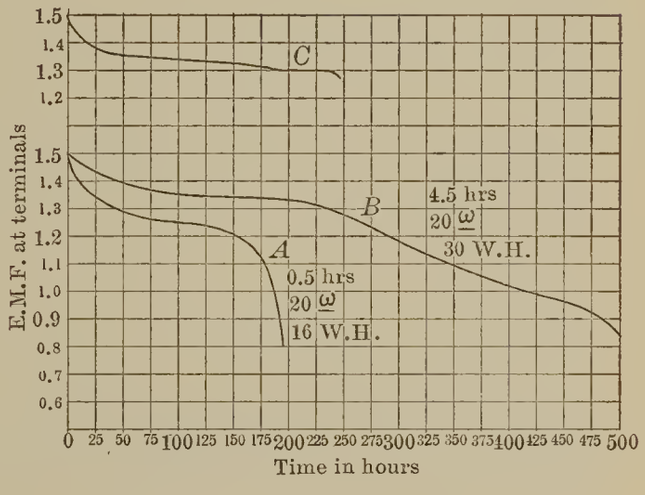


Fig. 5.

shown in curve D on Fig. 3. The conditions being slightly different from that of curve D, the two curves should be compared only with each other.

The integration has been carried to 0.9 volt and shows 8.5 watt hrs. for the longer duration (curve B), as compared to 9 watt hrs. for the shorter (curve A)—the energy, however, in these two cases may be considered as practically identical. We see here the same characteristic of longer duration and higher level with increase in resistance in the external circuit.

In Fig. 5 are shown two curves representing intermittent tests, which are similar to those on Fig. 3, with the exception that in curve A the resistance has been increased to 20 ohms. The duration of the test is approximately one year, and, considering this factor of time, the energy output is very good, the watt hours

being about 16 when figured to 0.9 volt. The current drain being light, it is probable that there is small opportunity for irregular action.

Curve B is for a still lower current, the resistance being increased to 25 ohms. Although the current is low, the time of closed circuit per day is comparatively long, thus bringing the watt hours up to the comparatively high figure of 30.

In integrating curves of this nature, the true value of course lies as a mean between the potential difference at the beginning of the closed circuit period and the potential difference at the end. In these integrations, however, the potential difference at the end of the period has been used because the desire has been to show characteristic action rather than to give exact laboratory data.

Curve C on Fig. 5 represents the result of a test made on ignition service. As no attempt was made to determine the energy, no deductions of this nature can be made. It is, however, of interest as showing what seems to be the requirement for this class of work, and that is a sustained high potential for the period of its useful life.

There are difficulties to be overcome in arriving at any adequate idea of the energy requirements for ignition service. The resistance as well as adjustment of spark coils is very variable and the time of service, as for instance in automobile work, is apt to vary between wide limits.

In striving to obtain the highest efficiency, it is necessary to know working conditions, such as current drain or resistance of circuit, and the frequency and duration of circuit closure. Knowing these factors, the proper arrangement for the required number of cells can be made to obtain the best results. It is frequently found that cells in parallel may be advantageously employed for the reason that excessive current drains may thus be avoided. The characteristic action of cells indicates that under heavy current drain the fall of potential is rapid and the energy obtained is small, hence the plan of placing *"enough"* cells in series may not be advisable; an excessive current is of no benefit and on the other hand draws energy in an inefficient way. A high potential may be maintained by placing cells in parallel so that the current drain on each will be light. Such a plan utilizes the energy

available in a far more efficient way, since we know that at high levels we obtain the greatest amount of energy.

Tests are sometimes made in which a fixed current is caused to flow, but in order to accomplish this, the resistance must be constantly changed. Such tests are interesting, but the conditions are not those ordinarily met in practice. A constant current flow is desirable, however, and a very close approximation to it may be made with dry cells, provided they are arranged for the high level rate of discharge.

One of the important factors in obtaining data from dry cell tests is the "cut-off" point, or, in other words, the value to which the potential difference of the cell may be allowed to fall before it is considered exhausted. The lower a cell is run, the greater will be the energy obtained, but this is offset by the greater variation in potential difference. Hence a compromise must be made. A study of conditions must govern the selection of this point in various classes of service, but it should be remembered that it involves one of the most important elements in obtaining best results.

The battery gauge is a factor in the situation that must be considered. As a rule, these instruments are constructed as a species of volt-ammeter, in that they are either high resistance ammeters or low resistance voltmeters. As they are not expensive, it can hardly be expected that they will compare favorably with Weston instruments in exactness of indications. The scale divisions generally indicate amperes, and the intention is to show what current would flow if the cell were short-circuited through a low resistance ammeter. The coil of the battery gauge being of only a few ohms resistance, there will be a variation in current depending on the internal resistance of the cell—the deflections of the needle will be approximately proportional to the current strength. When the internal resistance has increased to a considerable extent, the battery gauge is capable of giving a good indication of it. Thus when a cell is exhausted, a battery gauge is convenient and fairly reliable for indicating the condition of the cell. Battery gauges are not reliable for detecting small variations in the condition of a cell, nor for determining accurately any exact condition. Where exact conditions are desired, the best that can be done is to allow the normal working current of the

cell to flow and then take a potential difference reading with a standard high resistance voltmeter. Such readings give the best indications of the stage a dry cell has reached in the period of its useful life.

In conclusion, I wish to express the hope that before long some standard tests may be proposed which will be generally accepted as determining the rating of dry cells. The number consumed annually is enormous, and users are obliged to accept them with no information which will give a definite idea of energy output. They must judge of this by experience. The art of manufacturing dry cells has progressed to such a point that a reliable product is obtainable, and dry cells should fall in line with other electrical products as standard articles with a definite rating.

DISCUSSION.

PRESIDENT BAEKELAND: The dry cell, modest as it may appear as a unit, forms the base of a more important industry than most of us imagine. The number of dry cells consumed yearly can be counted by millions, the industry is more important than many electrochemical industries of which we hear much more in our meetings, and read much more in print. It so happens, at the same time, that it is one of the subjects on which a very large amount of misinformation is abroad, misinformation sometimes sent out purposely, but most of the time involuntarily through pure ignorance. It is almost impossible to tell beforehand whether a dry cell is good or not, because here everything goes by experience. In fact, I would almost say that the test of the dry cell is to use it for a particular purpose. We are familiar with the phrase "the test of the pudding is in the eating of it," but the trouble is that after the eating of the pudding there is none left.

I am glad to see that this meeting brings us several able papers on the subject of dry cells.

A MEMBER: What particular cells were tested in this work?

MR. LOVERIDGE: I had not expected to bring out the characteristics of any particular cell. The curves, as I have shown them

on the blackboard, are intended to represent characteristic action and they show about what results may be expected from cells made in accordance with what I believe to be recognized as perhaps standard practice at the present time.

MR. CLAYTON H. SHARP: I wish to express myself as seconding the recommendation which has been made that a standard series of tests for dry cells, the series to represent the different uses to which dry cells are put, should be adopted. As it is now, requests come to us sometimes to test a dry cell, or a set of them, and we have to say, "What kind of a test do you want?" The reply is, "Just an ordinary test, we want to know what the cell is good for. That still leaves the question indefinite and unsatisfactory.

In making such tests it has been our practice to try to reproduce as nearly as possible some of the conditions of operation of the cell, without at the same time making a test which would extend over an excessively long time. We have arranged a mechanism driven by an electric motor, with contact brushes so that the cell is put upon closed circuit with a definite resistance during a definite interval, and then thrown on to open circuit during another definite interval, these intervals bearing a known relation to each other. Then, by varying the ratio of the open and closed circuit intervals, the relative qualities of the dry cell under different conditions of service are indicated. One of these tests may be made under conditions where the load is the primary of an ignition coil.

If methods of this kind could be standardized, so that the ratio of closed circuit time to open circuit time is determined for different classes of service, it would be of very great service, I am sure, to those who are engaged in testing such apparatus, and would go a long way, perhaps, towards increasing the general knowledge of what dry cells will do and in improving the quality of the product which is furnished to us by the makers.

PROF. C. F. BURGESS: In considering the various methods of testing dry cells, it seems to me that consideration should be given to the test in which the cell is delivering a constant current. In the tests which have been described a constant resistance is employed and both the current and the voltage are variables. It

is true that in many uses the cells are connected up to some constant resistances, and consequently the current decreases from the time the cell begins service, but for convenience in testing, in comparing one series of tests with another series, and getting the data on a comparable basis, it seems to me that the cell should be discharged at constant current. Where this is done a number of different makes of cells can be placed in series, and simultaneous tests made on all of them with very simple equipment.

While most of the practical uses of the dry cell are those in which the cell is connected to a constant resistance, there are many uses in which the current is kept practically constant, or that is the intention. In ignition service, it is well known that a new set of batteries is given different adjustment to that which is given after the cells have worked for a while. The coil is readjusted so as to allow the current flowing to be maintained.

Further, the previous speaker has pointed out that it is necessary to discard a cell before it has reached its ultimate possible usefulness. Perhaps there is still thirty per cent. of the original energy in the cell when it is discarded. Now, there are some uses for a dry cell in which one additional cell can be put to the series from time to time, thus maintaining the proper voltage and keeping the current fairly constant again. In my experience I have found that a constant current discharge is preferable to one in which the resistance is kept constant.

Mr. Roger C. Aldrich: With reference to testing dry cells, it seems to me that a very important test is one which shows the life and capacity of the cell when current is taken from it in very small amounts at infrequent intervals; for instance, half an ampere of current for 5 minutes taken once or twice a week, and also the same volume of current for 5 or 6 hours at intervals of 3 months. The reason for this test is as follows: A large proportion of the automobiles on the market at present have magneto ignition with a set of dry cells for easy starting and reserve. These cells are thus used only a little, but ought to stand up a long time. This test would also apply fairly well to the case of a car with storage battery ignition, with dry cells used only as a reserve and when the accumulator is being charged.

In view of the present condition of the automobile industry,

which must use a great number of dry cells, there would be a large demand for cells which would stand up well under a test such as that just given, as this test would show, as nearly as possible, the value of the cell under actual working conditions.

The use, for a reserve, of the dry cells at present recommended for automobile work, is rather a joke, as if called on, when a few months old, for much current, there is a good chance of their being "tried and found wanting."

MR. SHARP: In answer to the remarks of Mr. Burgess, it seems to me his argument in favor of the constant current test is largely that it is a more convenient test to make. There is a little technical advantage in making a test in that way, because there is perhaps less trouble in putting the ammeter in the circuit, etc. That does not seem to me as really a very valid argument that the constant resistance test is a better test, or either that it corresponds more nearly to the conditions of practice. It is true that you can in practice add additional dry cells to a series, so as to maintain the proper amount of current or you can re-group your cells, but, as a practical matter, is it not true that a dry cell has a certain "useful life," as does an incandescent lamp, and that when it has passed a certain period of its life it is better to throw it away and get another dry cell, than to try troublesome expedients to recover the slight remaining amount of energy which it contains? I think it is cheaper to buy new cells than to bother with the old ones. After they have reached the end of their "useful life" they are not deserving of any further consideration.

MR. CARL HERING: It is suggested that rules be formulated for testing dry cells. I have long held that this would be very desirable, and it seems to me that this Society is the proper one to undertake the formulating of such rules. If it is in order, I make a motion that the Chair appoint a Committee to take up this question of preparing standard methods of measuring dry cells. The American Institute of Electrical Engineers have taken up similar questions in their field, and I do not see why we should not do so in ours. With such men as Prof. Burgess and Dr. Sharp on the Committee, although they seem to differ on some points, I think it would be possible to formulate quite satisfactory specifications for testing dry cells.

THE PRESIDENT: Gentlemen, you have heard the motion as made by Mr. Hering. It has not yet been seconded.

MR. W. R. WHITNEY: I second it. It seems to me that the users of dry cells in this country have the right to expect the American Electrochemical Society to do just this kind of work for them, that is, to outline in some way some system of tests, so that people may learn something about such materials as the dry cells, of which we use a great number. I have an automobile, I use some dry cells, and I find the conditions are not exactly those outlined by Dr. Sharp, that it is not a condition practically comparable with that of the incandescent lamp. The incandescent lamp does not deteriorate when not in use, whereas the dry cell deteriorates when not in use, and therefore it seems to me that this Committee which is being suggested, might find that they had to outline two courses of tests, one course to benefit the man who wants to use a set of batteries in his automobile, or some such place where the cells are being used only at the moment of starting the machine, and if he does not forget it, are immediately turned off. These batteries ought to last, judging from the chemistry of batteries, for a few years. As a matter of fact, they last only for a few months. In another case, a man may wish to operate bells or other apparatus continuously, and wishes to take energy out of the cells at a certain definite rate. For his benefit a different test should be made and probably different kinds of batteries could be found preferable in the two cases. This is a matter of interest to us, and it seems to me that we are the people to whom others have a right to look for some information and light.

DR. ARTHUR H. ELLIOTT: This question of dry batteries is one I have had some experience with, in use. The various kinds of batteries on the market today are as different as the birds of the air. It is largely due to the structure of the battery, the materials in the battery, and it is extremely important in one respect. For example, in the formula it says "ten pounds of manganese dioxide." Any one that knows anything about manganese dioxide knows it is very variable, and there could not be too much importance put upon that part of this question. When the Committee is looking into this matter, they should look into the question of the quality of materials entering into the composition of

the battery, and that will make a big difference as to the life of the battery and the actual useful efficiency derived from the battery.

Mr. Loveridge: I am very glad, indeed, to see a move made towards the appointment of a Committee to reach some standardization of tests on dry cells, and I hope that conclusions may be reached which will be satisfactory and acceptable both to manufacturers and users. One of the speakers has said that in making tests of dry cells an effort is made to reproduce conditions of operation. I believe he has adopted the right plan in doing this, but the limitation he places on tests in not extending them over "an excessively long time" might be open to objection. There are two factors to be considered in making tests of dry cells. The first is the electrical energy available for doing work in the external circuit, and the second is the "internal action," which so changes the conditions in the interior of the cell that the flow of current in the external circuit is either diminished or entirely prevented. The "internal action" is proceeding at all times to a greater or less extent, no matter whether the cell is giving out energy or not. No test is of value which does not take these two factors into consideration. It is easy to take a dry cell and make an accurate statement of current flow and voltage or to give energy output in watt-hours, but such results unaccompanied by a statement of rate and manner of discharge give no adequate idea of what may be expected under any definite requirement.

The second factor is really the one which determines the quality or excellence of the cell and the electrical energy available under any given condition of working. When cells are placed in storage before being connected in circuit, the "deterioration" or "internal action" is slight at low temperatures. As soon as there is a current flow "internal action" takes place which we may say is largely utilized as external energy. When the current ceases to flow "internal action" will continue, but to a much less extent. The volume of current flow in the circuit in which the cell is placed appears to bear a proportional relation to the rate of "internal action" that takes place when the current ceases to flow, and there also seems to be a tendency for this harmful "internal action" to continue at rates which correspond to the rates at

which it is started. I consider that this explains why cells in automobiles sometimes fail as "reserves" after a comparatively short time. When they are used the current flow is heavy and the internal equilibrium is disturbed to a corresponding degree, thus favoring rapid "internal action" or deterioration when not in service.

In this connection it might be stated that the "internal action" which takes place when no current is flowing differentiates the dry cell from the incandescent lamp, for in the latter case there is no "deterioration" except when the lamp is in actual service.

To sum up this phase of the question there would seem to be only one thing to do; and that is, to take the factor of harmful "internal action" into account by extending the test over a period that meets practical conditions in this respect.

With reference to the "constant current" test mentioned, I take it that the speaker means an "intermittent" and not a "continuous" test, as the latter would only extend over a very short time or else would be at an extremely low rate of discharge. In either case practical conditions would not be represented.

Dry cells are generally used where there is a definite number in the set. In some cases it is impossible to add to the number so that additions in series to keep up the current is not a method generally applicable. The maintenance of a constant current by the addition of cells would involve a closer supervision than is generally given to such batteries and the danger of exhausted cells remaining in the circuit after they had become "dead" resistances would also have to be reckoned with. Attention might also be called to the fact that the utilization of the total energy of cells would lead to an indefinite increase in their number. Hence the result would be a compromise with the cut-off point at less than the actual total of available energy. As pointed out by another speaker, the possible convenience of this form of test would seem to be the principal thing to commend it.

A paper read at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909. President L. H. Baekeland in the Chair.

POWER FOR ELECTROLYTIC COPPER REFINING.

By W. L. Spalding.

Power is an important item in the cost of electrolytically refining copper. Per ton of product, it is less than in many electrochemical industries, since with soluble anodes the voltage factor of the power is used mostly in overcoming the ohmic resistance of an electrolytic and metallic circuit and meets no opposing electromotive force from decomposition of the electrolyte. Nevertheless, this item is in round numbers 40% of the cost of converting anode to cathode copper or 20% of the cost, including furnace treatment. Its relative size, though not its actual, is increased by the extensive use of labor-saving machinery and mechanical handling of material. This use has been so far perfected that further reduction of costs must most easily be obtained through lower power charges.

In securing cheap power, there are several considerations which greatly aid the refiner. First, the unchanging resistance and the continuity of the process allow a constant load day and night, and the steadiness and magnitude of the power used insures the economy either of its generation at the plant or of its purchase from a power company. Secondly, waste-heat boilers on the reverberatory and cupola furnaces furnish considerable power at little expense, since the item of fuel is entirely eliminated from its cost, and were there a sufficient amount of it, the refiner would not need to seek further. Its one disadvantage is its irregularity; for several hours during the day, the furnaces are being dipped, charged or repaired, and furnish little steam, while for a couple of hours in the morning during poling they give a large quantity. The amount of this power available for electrolytic work depends upon the material treated at the refinery; one commencing with pig copper or anodes, as is usual in the East, receives relatively a much smaller amount than one smelting ores or concentrates.

Power for electrolysis may be either generated by steam engines, gas engines, or hydraulic turbines, or purchased from a power company. Steam engines, because of their reliability, are most commonly used; any available steam from waste heat boilers may be used in them directly by connecting these boilers in parallel with the coal-fired boilers, or the former may be used as preheaters and connected in series with the latter. Both reciprocating engines and turbines are met with, but the former more frequently, because of their high efficiency at full load, the availability of floor space at a smelter, and the lower speed which allows them to be direct-connected to the D. C. generator.

The high thermal efficiency of gas engines suggests, as an attractive possibility for a refinery, a central battery of gas producers, which furnishes both power for generating electricity and fuel for heating the furnaces.

The purchase of electric power from a power company is very desirable electrically, and under some conditions is the most economical. If A. C. power is received, the units for supplying the tank house circuits are synchronous motor-generator sets, which give a high efficiency of conversion, a 100% power factor, great facility of operation and regulation, and practically uninterrupted service. A refinery thus equipped should be the most favored customer of the company, as with proper regulation, the load curve is absolutely flat, and no interest charge on machinery held in reserve can equitably constitute a portion of the rates. Moreover, the refinery may be used for filling up the valleys in the company's load curve and the refiner may be able to contract for additional blocks of power, at such times as the company directs, for a figure lower than for his steady twenty-four hour load. It is true that the current density will increase at these times, but as the deposition of copper can proceed satisfactorily under a wide range of current density, this fluctuation is not open to the same objections as in processes that require a fixed minimum voltage or a constant supply of heat. A calculation of the saving from this cheaper power should involve the consideration that the cost of power per ton of copper is nearly proportional to the current density, and too great an increase in the density will overbalance the lower rate. On the other hand, steps may be taken to materially lessen this increase. The tanks with insoluble

anodes used for depositing excess copper from the electrolyte may be placed in circuit only at these times; each takes about eight times the energy of the ordinary tank, due to electrolytic decomposition, so that if their number is 2% of the total, their resistance is 14% of the total. A certain number of other tanks are also regularly short circuited during the day for cleaning. Also it must be borne in mind that the current density is proportional to the square root of the power, and hence its variation is less than that of the power.

In Buffalo, the cost of Niagara Falls power in lots of 500 H. P. or over is $27.50 per horse-power year, which, converted to D. C. at 87% efficiency equals $31.60, surprisingly large for a region situated near a source of vast water power. Unfortunately for the consumer, there is but one company owning a franchise in the city, and it has been able to keep the field free from rivals, and itself from adverse legislation. The price of power is, therefore determined not by competition with other power companies, nor by cost of production, but by competition with steam power.

The refiner who wishes to use both waste heat and electric power for electrolysis is confronted by the interesting problem of combining the two so as to secure minimum cost per ton of output. The most satisfying way seems this,—to supply a tank house circuit with two generators in parallel, one driven by a motor running on a constant amount of the purchased power, the other driven by a steam engine whose load varies with the steam.

This arrangement permits the load to be divided in any ratio between the two generators by hand or automatic regulation of the shunt field rheostats, or the engine to be shut down on Sundays and during the dipping of the furnace charges when the supply of steam is not more than sufficient for driving air compressors, pumps, etc., and heating or evaporating the electrolyte. At large ratios of the two loads the equilibrium is delicate, but not disturbed. When taking the load off both machines, if the rheostats are not turned simultaneously, one may overpower the other, but as the direction of rotation of a shunt-wound generator is not changed when run as a motor, no harm is done.

A series arrangement of the generators with half the circuit interposed between them would divide the voltage between the

tank house terminals and decrease the current loss through grounds, but excessive sparking from commutation of the heavy amperage in the weak field would not allow the engine-driven generator to carry as small a load at periods of low steam.

It is not economical to use the waste heat power on an independent circuit, as part of the time it would receive little or no current; the output of copper with a given power is proportional to the square root of the number of tanks and the electric power could be used to greater advantage if spread out over this circuit.

To determine the number of K.W. to be bought for each month, a calculation of the amount necessary for the desired production should be made, the probable power from steam deducted, and the remainder maintained as a constant load on the motor. When the power paid for is the maximum two minute peak occurring in the month, and these peaks are recorded by a graphic wattmeter, close regulation is essential. This can be obtained by hand regulation aided by a bell alarm, or, better still, by an apparatus that automatically changes the resistance of the shunt-field circuit of the generator when the wattmeter on the motor circuit records more or less than the desired quantity. With this attachment, the maximum two-minute variation from the average will be less than 5 K.W.

This combination of power necessarily gives a current density greater at night than at day, but the fluctuation is not objectionable and can be decreased as already mentioned. Further uniformity can be secured by using the peak of the steam for storing up heat in the electrolyte at night, and by rearranging the schedule of the furnaces so that their periods of dipping do not coincide.

The practicability of the use of electric power for refining copper has a direct bearing on a problem that is receiving considerable attention in the industrial world,—the problem of meeting, on the one hand, the demands of the electrochemical manufacturer for cheap power, and, on the other, the desire of producers of electrical power to obtain customers who maintain a constant load, or who, better yet, can use large amounts of it at times when the rest of the producer's load is light. The magnitude of the scale on which electrolytic copper refining is now conducted

makes the availability of cheap power an important consideration in the location of the refinery; and at the same time such a plant is an ideal customer for the power company. The existence of these common interests makes certain a mutual profit from their mutual recognition.

University of Buffalo,
October, 1909.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909, President L. H. Baekeland in the Chair.

THE MANUFACTURE OF AIR-SALTPETER BY THE PROCESS OF THE BADISCHE ANILIN- AND SODA-FABRIK.

By Dr. Schönherr.

Nitrogen is peculiar among the commoner elements in that at ordinary temperature it shows little inclination to enter into combination with other substances. Practically all the nitrogen in existence, therefore, is to be found stored up in the free state in the surrounding atmosphere, while its companion there, oxygen, is met with all over the earth in hundreds of combinations, as a component of nearly every rock and as the chief component of water. Combined nitrogen, on the contrary, is found in but few spots on the globe.

Does it not then appear wonderful, that an element so slightly inclined to form compounds with other bodies should play so important a part in the organic world? Wherever there is life we find complicated substances containing nitrogen as carriers of this life. The protoplasm of the plants, the albumen of the animal world, that is, just the substances in which the actual processes of life take place, are compounds of nitrogen. It would seem, therefore, that organisms must have the power of taking atmospheric nitrogen up directly and turning it into organic bodies. As a matter of fact this is only to quite a slight extent the case; only low forms of life, certain kinds of bacteria, can so change atmospheric nitrogen that it can be assimilated by the roots of the higher plants. In this way a continuous stream of atmospheric nitrogen flows, as it were, into the plants and from the plants to the animal world, but in spite of all attempts no one has yet succeeded in increasing this stream at will. On the contrary, if we wish to carry on intensive culture, particularly of our grain crops, we must make use of manures containing nitrogen. It is evident that stable manure cannot increase the nitrogen reserve of the organic world, for its nitrogen content is due to

decomposed organisms, but also when employing the inorganic nitrogen manures, such as ammonium sulphate and Chili saltpeter, we are using up a supply which the organic life of earlier epochs has stored up for us. For ammonia is a product of coal; it is gained as a by-product in the distillation of coal, that is, from coke-ovens and gas-retorts. Much of the precious substance is still lost, but even if all were recovered its quantity cannot be increased at will for the very reason that it is only secured as a by-product in another industry.

The second, much more important nitrogen manure, Chili saltpeter, is found in the rainless plateaus of Chili in enormous beds, probably due to the decomposition of sea-plants. Its occurrence in Chili is unique; in several other places on the earth small beds of nitrates are found, it is true, but none of them have the importance of those in Chili, where the annual export approaches 2,000,000 tons. For the chemical industry also is saltpeter an important raw material, being used for instance in the production of all explosives. But by far the greater quantity, about four-fifths, is consumed by agriculture, which must in future give to the soil even more combined nitrogen if it is to produce grain sufficient for the ever-increasing population and avert famine. At no very distant period the Chilian beds will be exhausted and it can thus be seen that it is of extreme importance to be able to combine the nitrogen of the air artificially in order to give the roots of the plants as large quantities as desirable. This problem can today be considered as solved, for the combination of atmospheric nitrogen is already carried out on a large scale by two processes. The first process, in the development of which Frank and Caro have gained great credit, produces the so-called "nitrolim," the active constituent of which is calcium cyanamide. If finely ground calcium carbide is heated in an atmosphere of nitrogen it changes into calcium cyanamide, taking up nitrogen and giving off carbon. By heating this cyanamide with water under pressure, the nitrogen is split off as ammonia. The same splitting off seems to take place in the earth, so that the lime-nitrogen can be compared in its effect as a manure with ammonium sulphate, but special precautions must be taken in its use. It cannot be employed for the many varied purposes to which Chili saltpeter is applied.

The second process gives a product which is in every respect the equal of Chili saltpeter, in certain soils indeed is even superior to it; namely "air-saltpeter," which has been placed on the market for some years under the name of Norway saltpeter or nitrate of lime. While the air in the preparation of nitrolim must be separated into its constituents, *i. e.* the nitrogen must be secured pure, in the manufacture of air-saltpeter the air can be used just as it is. The chemical reaction is extremely simple and the fundamental observations were made very early. Already towards the end of the 18th century, at the very beginning of our present-day chemistry, Priestley noticed that nitric acid is formed when electric sparks are passed through the moist air. Cavendish confirmed his observation, but at this early date no practical application could be made of it. In the course of his work on gas analysis in the middle of the last century, Bunsen noticed that nitric acid is also formed when oxy-hydrogen gas is exploded. These two observations teach us, therefore, that under certain conditions nitric acid is formed on heating atmospheric air. But, it will be said, we heat the air with every ordinary coal fire, with every gas flame, and notice no formation of nitric acid. There must be another condition present besides merely heating, which is not present in ordinary burning. This cannot be merely the high temperature, for we do not find any formation of nitric acid when using the oxy-hydrogen flame, which was early employed in industry and for lighting purposes; I will only mention Drummond's lime-light and soldering by means of hydrogen. In these operations nitrous gases are certainly formed, but in such extremely small quantity that they are not ordinarily noticed. The second point which must of necessity be observed if nitric acid is to be obtained in any quantity is a sudden cooling of the heated air. Thanks to the brilliant work of a number of scientists we can now follow by calculation the progress of the reaction which takes place on heating air and accurately define its conditions. Though I have spoken above of the formation of nitric acid, I must now express myself more precisely, for these labors have proved that nitric oxide is always the first product to be formed. Nitric oxide is a colorless gas composed of equal volumes of nitrogen and oxygen. If a mixture of oxygen and nitrogen is heated to a sufficiently high temperature, some of

this body is always formed accompanied by the consumption of heat. In the formation of 30 grams of nitric oxide, 21,600 calories are consumed. According to a general law, more is formed the higher the temperature is raised, but for each temperature there is a definite percentage above which the formation cannot be increased, however long the heating is continued. The bodies are then said to be in equilibrium. We must not think of this equilibrium as a stable condition as met with in mechanics. There, bodies in a state of equilibrium remain at rest relatively to one another; here, on the contrary, we must consider that in this state also the various bodies present react energetically on one another, but nothing of this can be directly noticed for the relative proportions of the bodies present do not change, because in a unit of time just as much nitric oxide decomposes again to its constituents as is formed in the same space of time. According to Nernst there can be formed in atmospheric air at an absolute temperature of

1500°	C.	.....	0.1%	nitric oxide
1928°	"	.....	0.5 "	"
2202°	"	.....	1 "	"
2403°	"	.....	1.5 "	"
2571°	"	.....	2 "	"
2854°	"	.....	3 "	"
3103°	"	.....	4 "	"
3327°	"	.....	5 "	"

We can see from this table that at absolute temperature of 1500° C. only 0.1 per cent. of nitric oxide can be formed. On raising the temperature by 428° C. only 0.4 per cent. more is obtained, *i. e.* 0.50 per cent., while on raising the temperature from 3103° to 3327° C. the quantity of nitric oxide rises by 1 per cent., that is about four times as much as for a similar interval at the lower temperature. In any case, however, it is evident that quite small quantities of nitric oxide are formed, so that a considerable useless ballast in the form of air has to be heated too.

When we calculate what quantities of heat must be used to produce at 1928° C. and at 3327° C. the same weight of nitric oxide, we find that they stand in about the proportion of 5 : 1, that is, we require five times as much heat to produce the same quantity of nitric oxide at 1928° as at 3327°.

From this it can be seen at once that those processes have most

chance of technical success in which the air is heated to a very high temperature. With the aid of the electric arc a temperature of 3000° C. can be attained, so that it ought to be possible to produce concentrations of 5 per cent. nitric oxide. This is unfortunately not the case. On raising the temperature not only does the absolute concentration attainable increase, but also the velocity at which the reaction proceeds: this reaction velocity, however, represents both the rate of composition and that of decomposition of the body, for in the same unit of time just as much of the body in equilibrium disappears as is produced. Thus the rate of decomposition also increases with the temperature, at 3000° C. it is about 10,000,000 times as great as at 1900° C., so that the cooling down must be effected with enormous rapidity. Strictly speaking the particles of air should pass through the interval of temperature from 3000° C. down to about 1500° C. in an infinitely small time, in order to preserve their original content of nitric oxide. This is, of course, impossible, so that the yield of any process chiefly depends on the more or less skilful manner of carrying out the cooling. Cooling by means of the excess of air carried along has always proved itself to be the most effective means, and this method is used, knowingly or unknowingly, in most of the processes applied technically. All other means of cooling work too slowly. The use of air as cooling agent is also favorable to the preservation of the nitric oxide formed, in that it reduces the concentration of the latter, for the rate of decomposition falls very quickly with the dilution.

These theoretical explanations of the reactions which take place on heating air, which are based on the idea of a purely thermal action, have been proved by a series of exact experimental investigations, but the recent work of Haber and König have made it doubtful whether they apply to electrical heating. Already at the general meeting of the Bunsen Society at Dresden in 1906, Warburg drew attention to the fact that in his opinion effects other than purely thermic were brought about in treating air in an arc, and later Haber and König succeeded by the observation of certain conditions in producing nitric oxide of such high concentrations as could never be obtained as the result of a purely thermodynamical equilibrium. In their experiments they worked with air at a pressure of about 100 mm. mercury, and obtained

concentrations of nitric oxide of 10 per cent. I am unable to go into these interesting experiments, but I would point out that these special results, which were certainly obtained by Haber using diffuse discharges, are so diminished in technical working when very large quantities of current are used with an air pressure differing only slightly from the normal, that the purely thermal explanation of the process can be retained for treatment under these circumstances.

The high concentration is dependent not merely on the temperature, but also on the proportion in which the two reacting bodies stand to one another. The equilibrium constant is found according to the law of mass:

$$K = \frac{C^2_{NO}}{C_{N^2} \cdot C_{O^2}}.$$

The concentration of NO has, therefore, the highest value at each temperature when the product $C_{N_2} . C_{O_2}$ is a maximum. It is directly proportional to the square root of this product. In air with 21 parts by volume of O_2 and 79 parts of N_2, this product is $0.21 \times 0.79 = 0.16$, while in a gas mixture of equal parts of oxygen and nitrogen it is $0.5 \times 0.5 = 0.25$. If this mixture is made use of instead of air, the concentration increases, and the yield accordingly, to

$$\sqrt{\frac{25}{16}} = \frac{5}{4},$$

i. e., by about 20 per cent. It must be thought that it would be profitable to make use of such mixtures rich in oxygen, but it is not yet done on the large scale. To produce such mixtures requires costly appliances and expenditure of energy, so that the higher concentration of nitric oxide would not represent profit. It would not be possible to let the air escape into the atmosphere after the last absorption, and lead fresh quantities to the burners, but a closed gas circuit would have to be provided and this involves many difficulties. At the same time I do not consider it improbable that in future such artificial mixtures will be worked with.

If the thermal explanation of the reaction is adopted, it is a matter of indifference of course in what way the nitrogen-oxygen mixture is heated, and consequently every imaginable method of

heating for this purpose has been proposed, as is shown by the patent literature of the last decade.

Oskar Bender (British Patent No. 8,653/07) heats the air in a generator by means of coal to a temperature as much above 1000° C. as possible. In the upper part of the burner is a tube through which is led superheated and strongly compressed steam, which is supposed to dissociate in the heat of the burner. The oxy-hydrogen gas thus produced causes, on burning, a further rise of temperature in the air which has been previously strongly heated. This is of course impossible, for the heat produced by the burning of the oxy-hydrogen gas must first be taken from the heated air. In the same way the Westdeutsche Thomasphosphatwerke try to heat a mixture of air and steam so high, that an extensive dissociation of the steam takes place. Before the gas mixture cools down, the hydrogen produced is removed by diffusion through porous walls, in order to avoid on cooling a reduction of the nitric oxide formed (German Patent No. 182,297). The diffusion is assisted by difference in pressure. Guido Pauling has patented a somewhat similar arrangement in America (Patent No. 758,774). He heats the air by means of an oxy-hydrogen flame and removes the small quantity of free hydrogen present by the addition of chlorine. Karl Södermann (Norwegian Patent No. 19,052) aims at previously heating the air in a system of tubes to 2000° C. and then raising its temperature by means of an acetylene flame to from 2400—2600° C. It can at once be seen, I think, that heating by means of acetylene, which must be gained indirectly from the calcium carbide produced by electrical energy, has no advantages over the use of electrical heating. Brünler and Kettler have taken out patents (France Nos. 363,617, 363,618, 380,467) for flames burning into water. Pawlikowsky (British Patent No. 26,728/05) proposes to produce the heat by compression, regaining the greater part of the energy expended by the following expansion. The heat used up in forming the nitric oxide he proposes to replace by flames from a second source of energy or electric discharges. Häusser (Verhandlungen des Vereins zur Beförderung des Gewerbefleisses, 1905, page 295) proposes to produce nitric oxide in a combustion compressor after the maximum temperature has been reached by means of compression and explosion, by spray-

ing in a cooling agent and so cooling the gases that a decomposition of the nitric oxide is no longer to be feared. The residual heat still contained in the compressed gases can be used in the production of mechanical work. The energy which is lost by spraying at the maximum temperature is to be taken as the cost of work in producing the nitric oxide. When the cooling is carried out for merely a short but fairly high interval of temperature, quite good yields should be obtainable theoretically according to this process. A somewhat different manner of carrying out the same idea is contained in Häusser's later British Patent No. 13,989/07. Here also he compresses combustible gases with an excess of air, but then explodes the mixture heated in this way in a special chamber.

Patents in which the heating is carried out purely by electric discharges are the most numerous, and up to the present only this method of heating has proved useful in actual practice. The reasons for this can readily be understood. On the one hand, by means of electric heating the highest temperatures at our command can be employed, which is very desirable for attaining high concentrations of nitric oxide; and on the other hand there is not the same possibility of carrying out the specially effective cooling by means of an excess of air with any other form of heating. With the other methods of heating, the concentration of nitric oxide is very low to begin with, and it is still further reduced owing to products of combustion becoming mixed with it. If then further quantities of air were added for cooling purposes the concentration of the nitric oxide would be so remarkably low that the difficulties of absorption, of which I shall have to speak later, would be enormously increased.

The first attempt to produce nitric acid by means of electric discharges taught that the yields were improved when arcs of a very small energy content were used. For a long time, therefore, it was held that discharges of a large current strength were to be avoided and the most complicated arrangements were devised in order to feed a large number of weak arcs from a single powerful source of energy. Every technical electrician can judge of the tremendous difficulties opposed to this way, and the exertions of a whole army of inventors were brought to nought by this problem of the division of energy. This is the reason why the

first attempts to produce nitric acid on a large scale by means of electricity, which were carried out in America by the Atmospheric Products Company, were not successful. The efficiency of the apparatus stood in no relation to its expensiveness. At that time the apparatus was described in all the technical papers, for the undertaking was the first of its kind and aroused everywhere the greatest interest. It consisted of an iron cylinder of 1.5 m. in height and 1.25 m. in diameter. In the axis of this cylinder a steel shaft rotated which carried 23 zones of electrode arms, each zone containing 6 arms and each arm being provided with an electrode. Opposite to these electrodes in the wall of the cover were a like number of fixed electrodes. The ends of all the electrodes were composed of platinum wire. The various rows of electrode arms were opposed to one another, so that during rotation as continuous a current as possible might pass through. In this way 414,000 small arcs were formed and extinguished per minute. The strength of the current was only a few milli-amperes per arc. Besides this, the apparatus was not fed with an alternating current, but with a direct current of 10,000 volts. The manufacture by the company, if the word manufacture can be used, for so far as I know only a single apparatus was worked, was stopped in 1904, as being hopeless.

A large number of patents has been taken out by other inventors also, in which weak arcs, or even spark discharges, have been used for the production of nitric acid. Many of these remained purely paper patents, others stuck in the experimental stage, so that I need not go into details on them. It was impossible for an industry on a large scale to develop on such a basis.

A sound basis for the industrial application of the combination of nitrogen by means of the electric arc was only obtained when Birkeland and Eyde succeeded in obtaining good yields with arcs with very strong currents. Their invention dates from the year 1903. They used the property of the magnet to drive an electric arc to one side, just as it will any other movable conductor. Their furnace consists of a low box-shaped space, lined with fire-brick into which copper electrodes cooled with water are introduced from the narrow sides. The furnace is arranged in the field of an electro-magnet so that the lines of force of the magnet run at right angles to the plane of the furnace. In conse-

quence of this arrangement an arc struck between the electrodes by means of an alternating current is alternately blown out upwards and downwards, and makes the impression on the eye of a large disk of light.

In this simple manner the Norwegian inventors created an extraordinarily efficient apparatus for the combustion of nitrogen, which worked without any moving parts and permitted of the application in one furnace of about one hundred times as much energy as did the complicated apparatus of the Atmospheric Products Company. There is now at work at Notodden, in Norway, a factory utilizing 30,000 h. p. according to this process, and the initial difficulties from which every new industry suffers are overcome.

Since towards the ends of the 90's at the instance of Dr. von Brunck, the far-seeing and enterprising manager of the Badische Anilin & Soda-Fabrik, experiments aiming at the artificial production of nitrate have been carried on in the factory at Ludwigshafen, utilizing the rich resources of the works there for the investigation. In the year 1905 I succeeded in discovering a method of burning nitrogen which requires an even simpler apparatus than the magnetic furnace. In working out this process I enjoyed the invaluable collaboration of my colleague, the engineer, Mr. Hessberger. In previous processes it had been regarded as advantageous if the arc only burnt intermittently, or on the other hand if burnt continuously then it was regarded as desirable that there should be very rapid vibrations through the air to be treated and finally in the Birkeland-Eyde furnace there were a series of rapidly moving arcs combining the two previous ideas. Now as distinguished from these processes, the process of the Badische Anilin & Soda-Fabrik makes use of an arc of great current density which burns quite steadily and is maintained in a stable condition.

At the time when the process was being worked out it was generally assumed that in order to obtain good yields of nitric oxide everything must be avoided which might lead to the air remaining for any considerable time in the sphere of action of the electric arc. The air was consequently always blown through the arc, for instance at right angles to the principal length thereof, and great stress was laid upon the value of preventing the air from

passing several times into the discharge and of removing the air as soon as it had once passed through it. Consequently all earlier processes for the combustion of nitrogen by means of stable burning arcs were characterized by the fact that the direction of the air current was practically at right angles to a line drawn between the two electrodes.

I departed completely from this theory and passed the gases which were to be treated *along* the arc. According to the theory then prevailing, the yields of nitric oxide should have been miserable in consequence of the oxide which was formed, being to a very large extent decomposed back again into its elements, and it was prophesied that the process would be of no practical use. These prophesies, however, were altogether wrong, as we shall see later on.

In my work on the problem of burning nitrogen, I soon learned that it was quite impossible to blow large quantities of air directly through an arc, for instance, at right angles to it. In such attempts only a very small proportion of the air goes into the sphere of action of the electric discharge and very dilute gases are obtained. Moreover the arc is very liable to be extinguished. On the other hand we obtained highly concentrated gases if the air was led along a quietly burning arc. This method of leading the air along the arc also imparts to it a much greater stability.

A freely burning arc of great length is, as is well known, an extraordinarily fragile thing. It goes out on the least provocation and especially easily when it comes into contact with cold air, but if it is desired to have good energy yields it is essential that considerable quantities of air shall be brought into contact with the arc, so that the current of air must possess a very considerable velocity. This, of course, tends to increase the danger of blowing out the arc. When the air is passed along the length of the arc, however, an irregular contact with cold air can be more easily avoided than is the case when the air is blown through it, and in consequence the arc burns more quietly and with greater stability. Our process can be most simply carried out by making use of a tube in the axis of which the arc burns and through which a current of air is passed along the length of the arc. It is not necessary that the whole quantity of the air be passed through the entire length of the tube, but the air can be passed in through

openings or slits which are situated at different parts of the tube, or they may be situated over the whole surface thereof. The cross-section of the tube is not necessarily circular; it may be angular, or oval, or it may possess any other form, and it is not necessary that it be the same at all points of the tube. For instance, the tube itself may be wholly or partly conical. The length and diameter of the tube may vary within very wide limits. All these variations, however, are of minor importance provided care be taken that the direction of the gas current be principally parallel to the burning arc. It is, in fact, essential that the electrodes and the current of air be so arranged that the arc is compelled to take up its position inside the current of gas. Our process is not necessarily limited to long straight arcs such as we have hitherto used on the practical scale. A continually burning electric arc can be obtained in the form of a spiral, if one electrode be situated on the periphery and the other at the center of a narrow space and the air be passed into this space in the form of a whirlwind from the periphery to the center. The arc follows the air current which in this case travels in a spiral.

The knowledge that the gas must pass in the direction of the length constituted a striking advance over the processes then in use, and we were able by using a special artifice to bring ourselves a long step further. We found, too, that the stability of the arc was considerably increased when the air was passed along the arc, not in parallel straight lines, but giving it a whirling motion so that as it passed along the arc each particle moved in a path resembling the thread of a screw. A direct current arc would possess many advantages for our purposes, but owing to the present state of electrical technical knowledge it is scarcely possible that these direct current arcs can be made use of. This, however, is now a secondary matter, since by giving a whirling motion to the air supplied, we are able to obtain by means of an alternating current an electric arc burning as steadily as the flame of a candle, and it can be enclosed in a comparatively narrow metallic pipe without the arc striking over to the wall; if the air be directed along the arc in a straight line there is a tendency for the arc to strike into the wall earlier than is desired, and when working in this way it is not easy to use a metallic pipe, whilst the use of a non-conducting tube has its disadvantages. In our manufacture

at the present time, we use exclusively arc flames burning in the center of a whirling current of air.

The apparatus itself is very simple. It is only necessary to arrange in a tube an electrode insulated from this and to drive through the tube a current of air giving it at the same time a rotary motion and then to start the arc in any suitable way. This can be effected, for instance, by making the distance between the insulated electrode and the wall of the tube at one place so small that, on open circuit, the voltage which is employed is sufficient to enable a spark to spring across. As soon as this has happened, the current of air drives one end of the short arc which is at first formed along the wall of the tube and the result is a long arc flame burning in the axis of the tube, the end of this arc striking the wall at a considerable distance from the insulated electrode. This point is determined by the temperature of the gases; at the place where the gases have acquired such a high temperature that they have sufficient conducting power for the arc to pass through them, this of course happens. As there must always be an inductive resistance in the arc circuit, when once the arc is started the electric tension sinks to such an extent that there is no occasion to fear the arc striking across again at the narrow part where the first ignition was effected. Should the arc go out for any reason, its re-ignition occurs automatically. This method of igniting the arc can be seen from the apparatus to the left in the accompanying sketch (Fig. 1). The tube which serves as the outer boundary of the whirling air current is in this case of glass. Within this can be seen a spiral of metal foil; it may be taken that this is a metallic tube-furnace, such as is used in manufacture, from which all the metal has been removed except this spiral. This must necessarily remain in order to permit of the arc climbing up the apparatus to the metallic head of the furnace. The end of this spiral at the bottom is only a short distance from the insulated electrode situated in the axis of the tube, so that at this point, when the tension in the circuit rises to 5,000 volts at first quite a small arc is formed. Later when the arc-flame is burning, the tension falls so much that no current passes over at this point and it can be seen that even higher up the arc does not touch the spiral, but only ends on the metallic head of the furnace. Of course all the conditions must be suitably chosen. The small

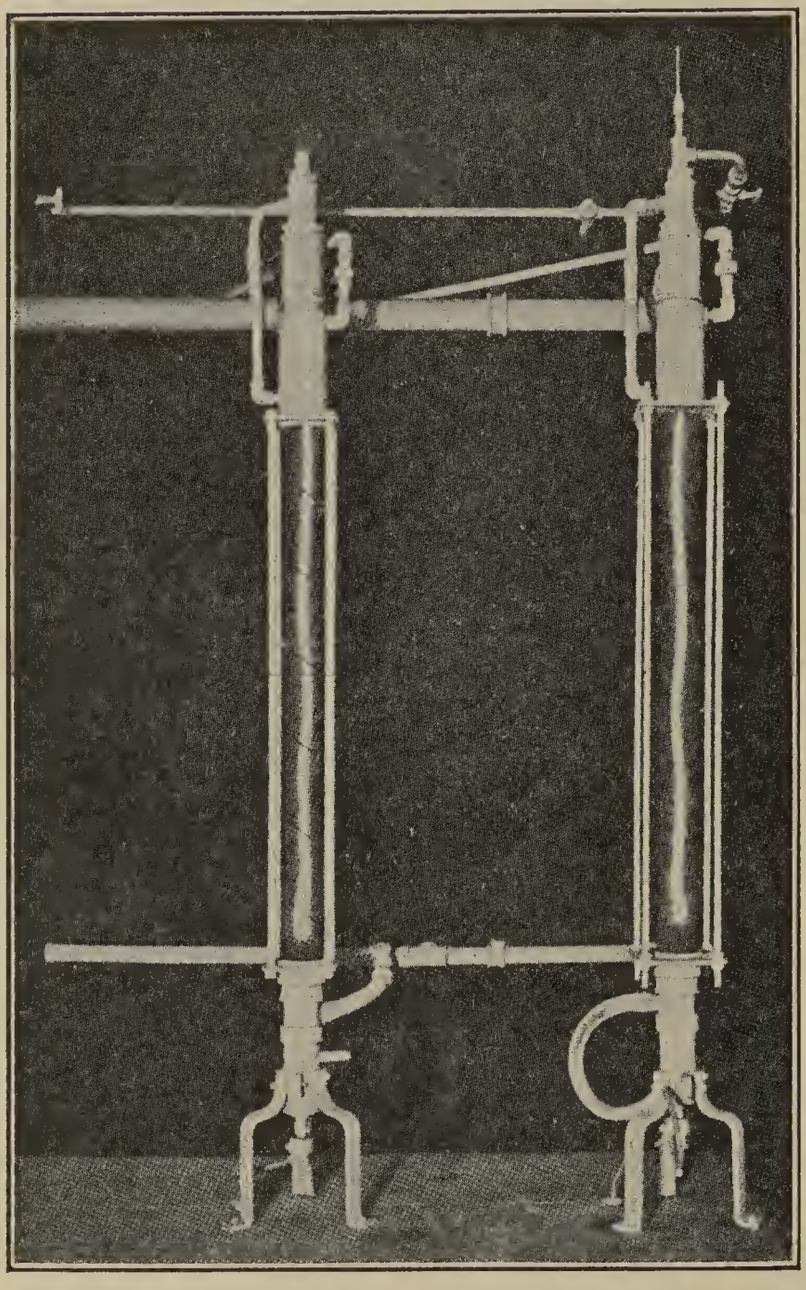


FIG. 1: Demonstration Tubes.

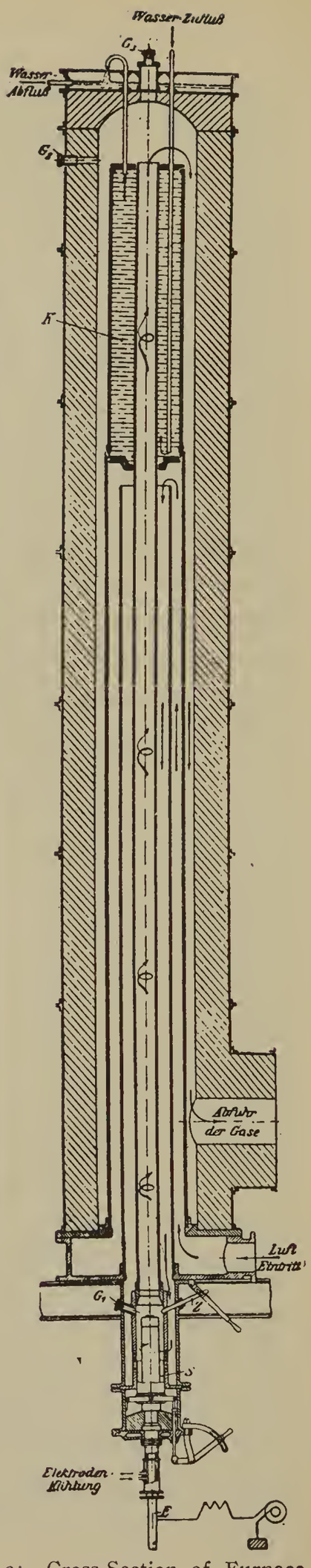


FIG. 2: Cross-Section of Furnace.

arc shown in this apparatus utilizes about 7 kilo-watts electric energy, that is to say in round figures 10 h. p.

This method of igniting the arc is very suitable for demonstration purposes, but it is not used in our actual furnaces. We abandoned this very soon because in manufacture it can readily give rise to interruptions. In the other furnace to the right in the Fig. 1 the spiral is omitted. In this case, in order to start the arc it is necessary for a moment to bridge over the distance between the two electrodes with the aid of a wire, and it is a method similar to this which is adopted in manufacture. In Fig. 2 I show a diagrammatic drawing of a furnace as used in manufacture. You will see on this a lever by means of which a massive iron bar *Z* can be moved from the wall of the tube, which for the time being acts as the second electrode, to the insulated electrode *E*. As the entire casing of the furnace with the sole exception of the central electrode is put to earth, there is no danger for the operator when igniting the arc.

In an experimental manner we have tried every conceivable method of igniting the arc in our furnace. It can be effected by permitting a spark to cross from the electrode to the wall, or by blowing in a small flame from an opening in the outer wall against the central electrode, or a very fine wire having great resistance can be passed from the wall of the pipe to the insulated electrode. Immediately the current passes, this is burnt up and passes away as dust on account of the great current density. A moist splinter of wood, or anything of that kind can be similarly used. As the electric tension amounts to 4,000 to 5,000 volts very slight conductivity is necessary in order to bring about the ignition of the arc. On account of its curiosity I will mention one more experiment that we have made. We have ignited the arc by firing a blank cartridge from a revolver at the inner electrode. These experiments were made in order to determine how far it may be possible to diminish the inevitable rush of electricity which occurs at the moment of ignition, but as our furnaces only exceptionally require reignition the method that is adopted is of small importance.

The rotary motion of the air can be obtained in the simplest manner by forcing the air into the tube in a tangential direction. If the air be blown into the tube through a single opening, its

course will naturally assume the form of a screw with one thread and the course of the screw forces the arc flame somewhat to one side, giving this to a certain extent the screw form, which can readily be seen from the photograph of the arc in Figs. 1 and 3.

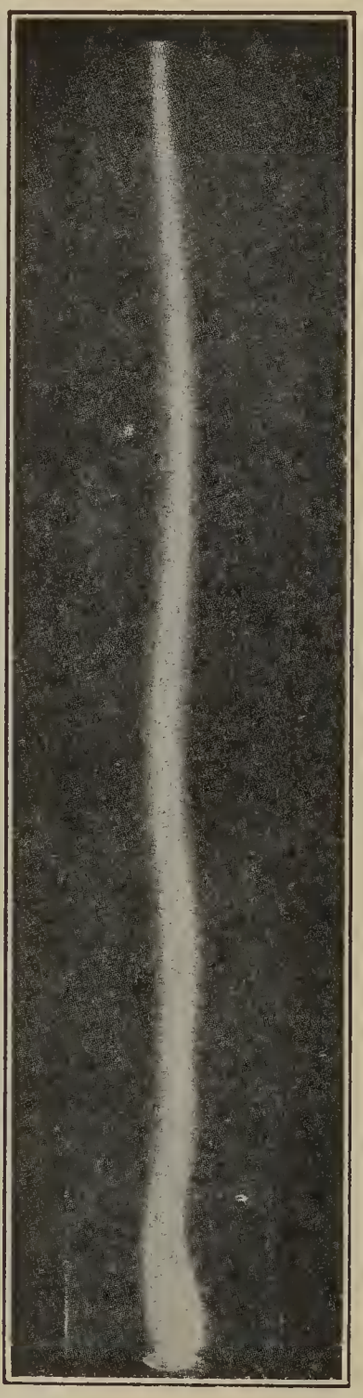


Fig. 3: Arc, 75 cm. Long, in Glass Tube.

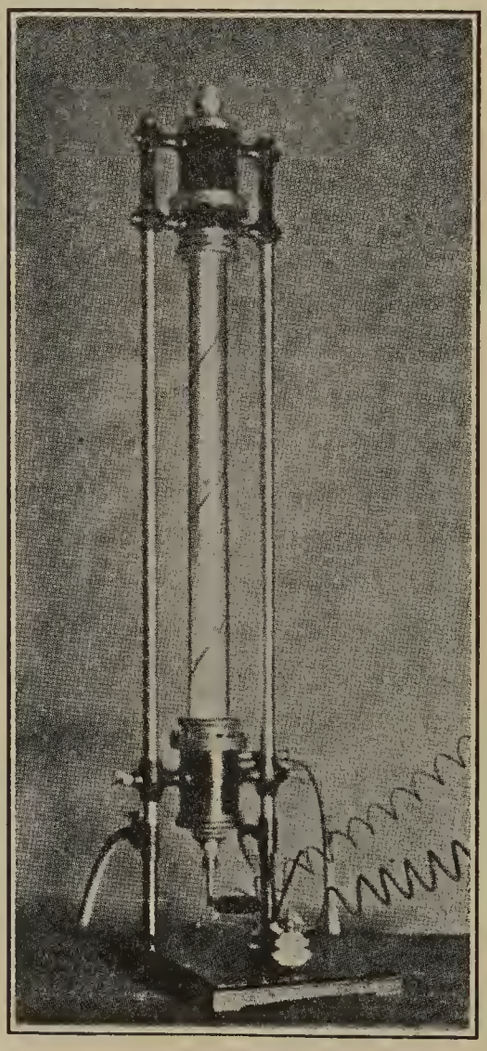


Fig. 4: Demonstration Apparatus of Newer Construction.

In the newer apparatus, Fig. 4, the entrance of the air into the furnace is effected through a series of eight openings in the same plane at the bottom of the tube. All eight openings are bored tangentially in the wall of the tube, and the air passes into the

tube in a very even current. In this furnace no deformation of the arc-flame occurs, it can be seen to be burning in a straight line. In the manufacturing furnaces the air passes in through several series of tangential openings the series being situated one above the other and over these a movable sleeve S can be moved. In this way it is possible, whilst using the same quantity of air, to lengthen or shorten the arc-flame. We can consequently feed the

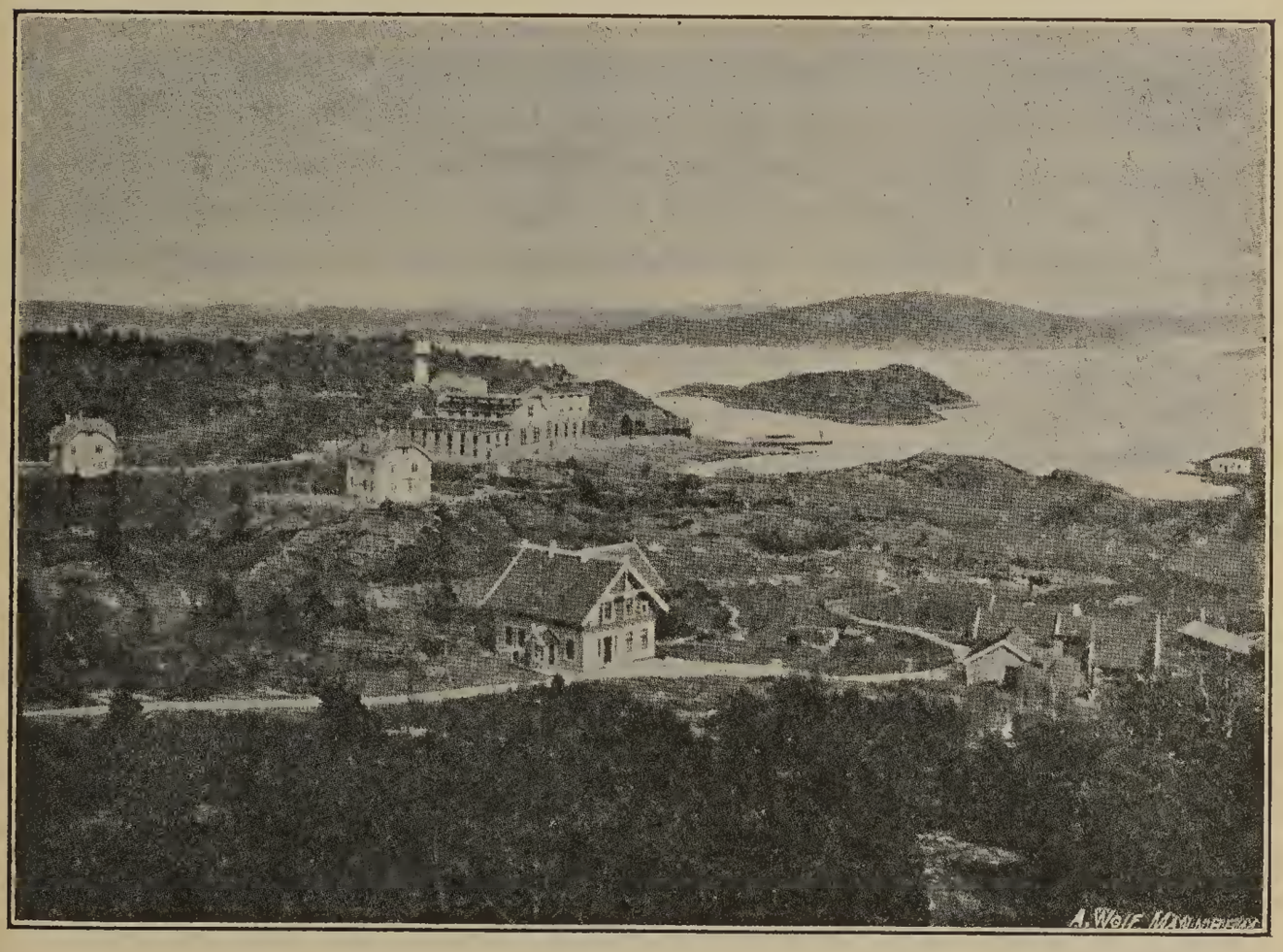


FIG. 5: Experimental Works Near Christianssand, Norway.

furnace with varying quantities of energy and still keep the length of the flame about the same and obtain gases of practically the same concentration of nitric oxide.

The equipment in Ludwigshafen only permitted the utilization of a single furnace fed with 300 kw. of energy. We therefore erected an experimental factory in Kristianssand in the south of Norway in order to test experimentally our furnace in continuous work, using larger energy units. This factory started working in the autumn of 1907. It worked well from the begin-

ning, and our furnaces after working for more than a year have proved themselves to be fully successful. The energy is obtained from a power house in the Saeterstal 26 km (16 miles) away, in the form of a 3-phase current. The transmission is effected with a tension of 25,000 volts with triangular connection. In the factory the current is transformed to one with 7,200 volts, so that the tension of each phase is 4,200 volts. The furnaces themselves are connected with star connections. We have available at the factory about 1,300 kw. in the form of this 3-phase current, so that we could feed the three furnaces with 600 h. p. each. The Figs. 5, 6 and 8—10 show views of this experimental factory. For short periods we have worked with furnaces utilizing 1,000 h. p. and our future factories are to be fitted only with this larger type of furnace. We have even thought we can build with equal success furnaces utilizing 2,000 h. p.

Fig. 2 shows the construction of our present furnaces. The electrode which is insulated from the rest of the furnace can there be seen in the axis. It consists of a strong copper body pierced through its axis and cooled with water. Through the axis passes an iron bar *E*, which can be pushed forward as required and constitutes the actual electrode from which the arc proceeds. All the wear comes upon this bar. Under the intense heat of the arc it becomes covered with a layer of melted ferric oxide, which slowly evaporates. As this evaporation proceeds the iron bar is pushed forward. This only occurs very slowly and the wear and tear of the electrodes is an altogether trifling matter. The costs under this head are only a few farthings per kw. and year. The bars of the length that we use for electrodes last for about 2,000 working hours, that is, for about a quarter of a year. To insert a new bar, it is simply screwed on to the old one, and this can be effected in about 15 minutes. The water-cooled copper body which surrounds the iron bar is kept at such a distance from the wall of the furnace tube that there is no occasion to fear current passing from it to the wall. The igniting rod *Z*, which has already been mentioned and which can be moved till it touches the electrode, is used for starting the arc. There is a peep-hole G_1 near the bottom of the furnace through which the starting point of the arc can be seen, so that the adjustment of the electrode can be effected at the right time. We per-

form this adjustment by hand as required, but, if desired, it could easily be effected continuously by purely mechanical means.

We have recently made use of cooled, hollow metallic electrodes. The arc springs from the inner wall thereof and by passing air through or round the electrode, or by other means, for instance magnetic influence, it is easy to cause the starting point of the electric arc to change its position continually on the electrode. The wear and tear of such an electrode is then reduced to a minimum.

In the figure the sleeves for regulating the air current, which has already been mentioned, can also be seen. In our Kristianssand furnaces, utilizing 600 h. p., the arc is about 5 meters long; in the 1,000 h. p. furnaces which were worked with a higher tension, arcs of 7 meters length were obtained. The tube enclosing the arc is simply an ordinary iron pipe. The arc only ends on this pipe for a moment when it is started, so that it wears practically forever. The upper part of the tube is constructed in the form of a special cooler, like a Liebig's condenser. The arc is made to end in this part of the apparatus by suitably manipulating the air-regulating valve. There are two peep-holes in the upper part of the furnace through which this can be checked. The opening at the top permits a view along the axis of the arc and looking down this way it can be readily seen that the upper end of the arc changes its place constantly, moving round in a circle, but the arc varies also slightly in length, so that the end of it comes into contact with a comparatively large surface of the cooler, and no part gets over-heated. The wear of the cooler is consequently very slight. Apart from this it is so constructed that it can be readily repaired. It is only necessary to roll in a new tube. We removed the tube of one cooler after it had been in use for four months, and it was hardly possible to notice any wear at all. After the hot gases have passed through the cooler they pass down an annular space surrounding the inner portion of the furnace, and from this enter a flue common to all the furnaces, and through this flue they pass along to a boiler which acts as a cooler for the gases and in which steam is generated that is subsequently used for concentrating the solutions produced later on in the process. It will readily be seen that the gases on passing down the annular space in the furnace give up a portion of their heat

to the tube through which the air supply passes to the furnace, as can be seen from the figure. This air enters the outer part of the furnace at the bottom, passes upwards, and then downwards, turning below the cooler, and in this way takes up heat from the gases passing away from the furnace. When the air enters the furnace it has in this way been subjected to a thorough preliminary heating. The entrance to the inner tube containing the arc flame is effected, as has already been mentioned, through the tangential openings.

It is not essential that the arc flame be vertical. We have also used furnaces with a horizontal flame, and this position can be shown by turning a demonstration furnace on its side. It can be seen that in this position also the flame burns quietly in the axis of the tube and the ignition can be effected in the same way as in a vertical furnace. Similarly, if desired, the air current can be passed in from the top to the bottom, and if the furnace be made short enough and the exit end of the furnace left open, an enormously hot blow-pipe flame possessing strong oxidizing properties can be blown out of it. An arrangement of this kind is specially suited for smelting operations, accompanied either by oxidizing or reducing effects, which are brought about by suitably choosing the gases employed. For such a purpose the melting crucible and its charge would act as the second electrode. Of course if desired several tubes can be used, for instance three tubes can be connected with the three phases of a 3-phase system, and the melting crucible and its charge would be connected with the neutral junction. Further, if desired, the arc flame may be made crooked; it may for instance be vertical in its lower part and horizontal in its upper part. This can readily be arranged with the aid of the whirling motion given to the air, in particular, after a bend in the furnace, a further supply of air can be blown in through tangential openings. It is of course in no case necessary to blow in all the air at one part of the tube; on the contrary it can be effected at several places lying far from one another, and in this way it would be possible, if it were desired to do so, to produce arc flames of very much greater length. Again, the tube which encloses the arc flame need not be cylindrical, it may be entirely or partly conical. All these modifications are mentioned to show that almost any desired manipulation of the

electric arc flame is possible; when using the whirling motion of the air as described the arc flame can be kept stable in positions of all kinds to a degree that appears hardly possible. When our furnaces are properly set up, it seldom occurs that the arc flame requires igniting afresh. Interruptions only occur if for some reason or other there are sudden violent variations in the air supply, or again if a short circuit occurs in the supply line, or

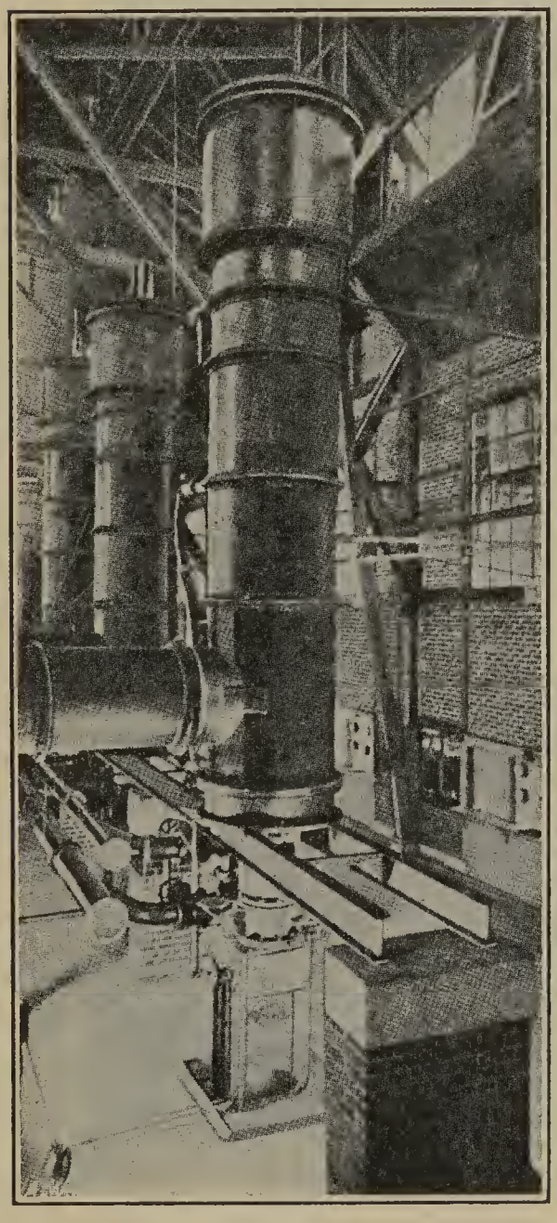


Fig. 6: Interior View of Works.

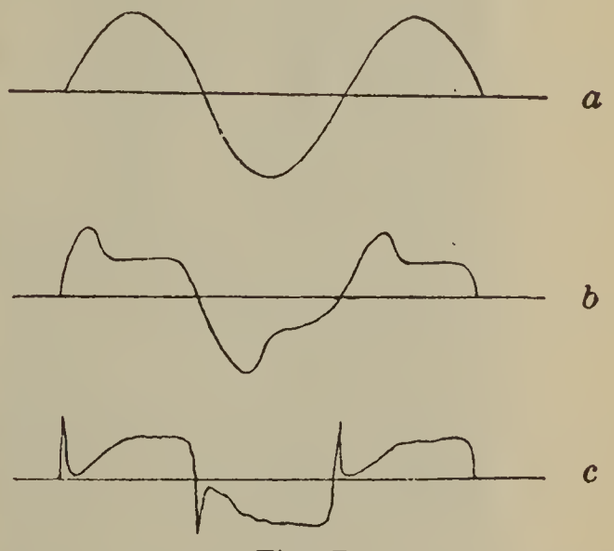


Fig. 7.

Fig. 7: Oscillograph Curves of Wave Forms

from any other reason the tension of the current drops essentially. The current and voltage curves can be seen to coincide nearly with the curves of sines in Fig. 7. Curve *a* is a chart of the voltage curve of our arc flame, curve *b* is the corresponding curve

from an ordinary electric light lamp, and curve *c* is the curve of the Birkeland-Eyde arc. It is noticeable that the ignition peak in the tension curve which can be most clearly seen in the case of the other arcs, is entirely absent in ours, and that there is only a very slight unevenness in the rising and falling part of the curve.

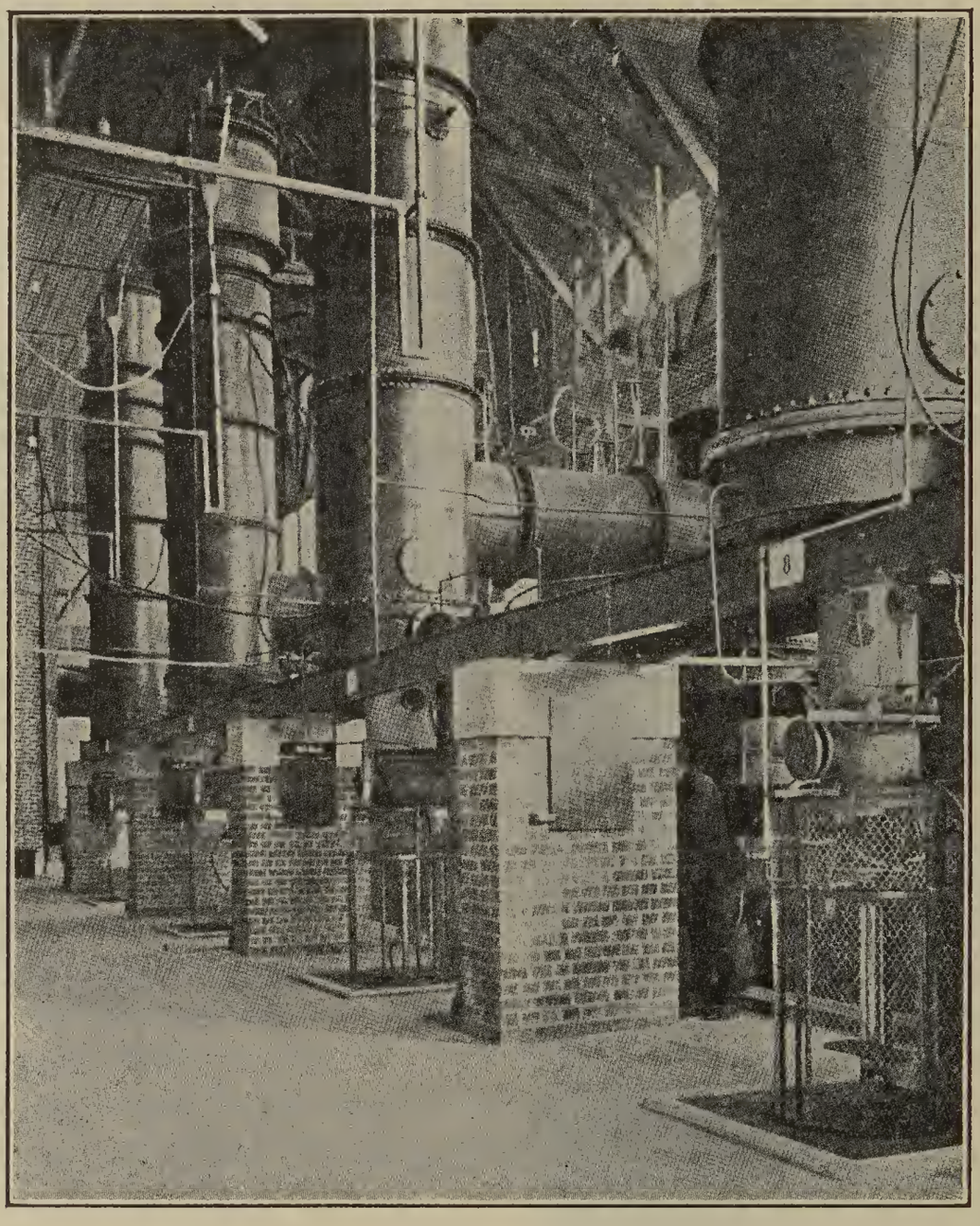


Fig. 8: Furnace Room of Christianssand Plant.

Agreeably with this the efficiency of our arc flame is very nearly 1 ; it amounts to from 92 to 96 per cent. This excellent result can probably be explained as follows. During several periods probably the current is conducted by practically the same air particles, consequently smaller variations in the resistance occur than is usually the case with electric arcs. Of course in every unit of

time new air particles enter into the field of the current and others leave the arc flame at the other end, but the number entering and leaving the arc is small compared with those which at one time are in the arc flame. An objection has been urged to our arc flame by a critic, that the air remains too long in contact with it, but from the above it can been seen that this is not a disadvantage, but in this respect distinctly advantageous. The fact that the air is acted on for a comparatively long time in the arc flame has no injurious effect on the yield either, for with our arc flame there can be obtained essentially higher yields than according to the process of Birkeland and Eyde. As the air particles which conduct the current do not mix with the rest of the air between the alterations of the current, no cooling of these particles takes place during these intervals, except by radiation, and this, as is well known, is very small for gases, so that it can readily be understood that the prolonged treatment of the air in the arc flame does not necessarily involve a loss of energy. Our tube furnace really corresponds in its action completely to the apparatus used by Nernst for the examination of the nitrogen-oxygen equilibrium. In this apparatus also the mixture of gases was heated for a long path, so that with certainty the equilibrium should be attained, then for a short portion of its path it was quickly and effectively cooled. In our furnace the cooler air which surrounds the arc flame like a mantle and which prevents the too early discharge of the arc to the enclosing iron tube, only becomes gradually warm. This air of course keeps at a temperature far below that of the arc flame itself, and at the point where the arc strikes through it, it serves in the most efficacious manner for cooling down the most highly heated air of the arc flame which is also that which is richest in nitric oxide. The whirling motion assists this cooling effect, in that it brings about a perfect and sudden mixing of the air and causes the flame to move so as continuously and regularly to change the position of its end point.

We have experimentally determined the fall of tension in the arc flame by the insertion of subsidiary electrodes, and have found that the fall in voltage proceeds fairly evenly along the whole length of the arc flame. Determinations of the concentration of the nitric oxide have also been made at different parts of the arc flame, and as was to be expected it was found that the

concentration was highest in the axis of the flame and towards the upper end of the arc. In the actual path of the arc flame itself, it is probable that the concentration is equally great all along, but owing to the higher velocity of decomposition and the tem-

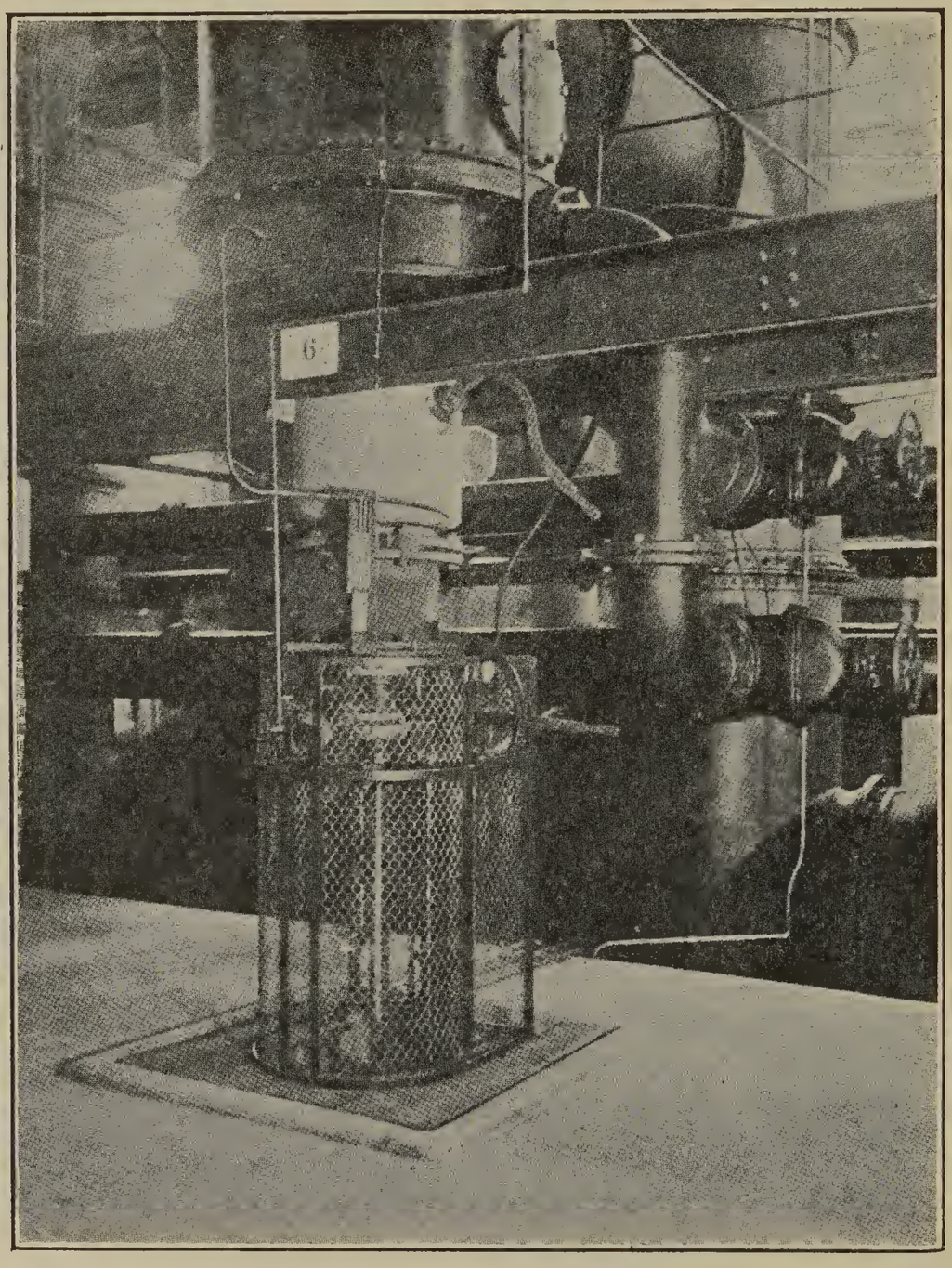


Fig. 9: Under Part of a Furnace.

perature of the arc flame it is unfortunately impossible to determine this concentration by taking a test sample directly.

Turning now to the question of the utilization of the energy, it must be confessed that if only that portion of the energy be taken into consideration which is used for the formation of nitric

oxide, this proportion is very slight, since only about 3 per cent. of the total energy that is fed to the arc flame is utilized for the production of nitric oxide; but this method of calculation is not accurate, for besides the nitric oxide, a quantity of highly heated air is obtained, though only a small quantity of the energy contained in it can be used for other purposes. It has frequently been attempted to calculate how far the process of electric oxidation of nitrogen can be improved. In these calculations a definite temperature to which the entire mass of gases is heated is assumed, and then the quantity of nitric oxide obtained at this temperature is compared with that which should be obtained according to purely theoretical considerations; such calculations are entirely empirical of course. They have only very slight practical value, for in none of the processes working with electrical heating is the entire quantity of gas brought to a uniform temperature and no exact figures showing the actual distribution of temperature in the gases are known, so that we cannot determine how far the yields obtained correspond to those theoretically possible.

It has already been explained that the air blown into the actual furnace tube is subjected to a preliminary heating. The degree to which this can be taken, is a pure technical question depending principally upon the durability of the apparatus. If the preliminary heating is effected in iron tubes, it is not well to go above 500° C. Now the best yields are to be obtained if the air which has been raised to a high temperature by the preliminary heating is directed to the axis of the furnace tube, so that it enters the actual reaction zone whilst this nucleus of heated air is surrounded by a mantle of colder air. This can readily be effected by the aid of our process by a suitable arrangement of the air supply, for instance immediately above the electrode we blow in hot air and higher up the tube cold air. The heavier cold air remains outside near the wall of the tube in consequence of the whirling motion.

If a balance sheet of the entire heat obtained from the furnaces be made, it is seen that in round figures 40 per cent. of the energy supplied to the arc flame is obtained in the form of hot water, 17 per cent is lost by radiation, 30 per cent. is used in the steam boiler, and 10 per cent. more has to be withdrawn from the gases

after they have passed the boiler by cooling with water, whilst only 3 per cent. is actually used for the production of the nitric oxide. The heat that is used for the production of hot water and steam could be utilized in other ways. The steam is used for evaporating the solution, so that no other source of heat is required in the manufacture except the electrical energy. It is to be expected that in this matter of utilizing the heat in the gases further advances will be made. We have considered, for instance, the idea of building our furnaces directly inside a steam boiler, but have not adopted this plan.

I have now described the main outlines of the process of the Badische Anilin & Soda-Fabrik. It can be seen that it utilizes very simple means. The furnace consists essentially of a system of pipes. They are cheap, reliable in actual manufacture, and last a long time. There are no moving parts and no expensive electro-magnets. The apparatus permits of the use of large units in manufacture and splendid yields are obtained. Besides this, this process furnishes the nitric oxide in a far higher concentration than most other processes. This is a point of great importance for the absorption, as to which I have still a few words to say.

We have seen that, for theoretical reasons, a sudden cooling must succeed the heating of the air. It is sufficient if this cooling is effected down to a temperature of about 1200-1500° C. At this temperature the rate of decomposition is already so low that it causes no further difficulty to remove from the gases the heat they contain without any further essential loss of nitric oxide. As a matter of fact the gases leave the actual flame tube of our furnace with a temperature of about 1200° C., and at the entrance to the gas flue common to all the furnaces they have a temperature of about 850° C. Directly the temperature sinks below 600° C., the colorless nitric oxide combines with a further quantity of oxygen taken from the excess of air present, whereby it is converted into nitrogen peroxide, NO_2, a gas having a brown-red color. Complete conversion only takes place below 140° C., and takes a considerable time. The velocity of reaction becomes slower the more nearly it approaches completion, that is to say, the smaller the concentration of the nitric oxide in the gas mixture; a mixture of gases containing two per cent. of nitric oxide

cooled down to 20° C. requires 12 seconds for the oxidation of half of the nitric oxide, but 100 seconds are required until 90 per cent. of the oxidation is effected.

It is necessary to absorb this nitrogen peroxide, the brown-red vapors that can readily be seen in the demonstration pipes, or rather in the bottles inserted into the pipe system for the purpose. It is not a simple matter to effect this removal of the nitrogen peroxide from the air. Alkaline liquids such as soda solution, milk of lime, and the like, combine with the gases forming equal proportions of a nitrite and a nitrate. This mixture can be used directly as a nitrogen manure. The statements that the admixture of nitrite is injurious to plant life are the result of a mistake. Von Lepel, Schlœsing and lately Wagner have made culture experiments which have fully proved that nitrite is not injurious to plants, and the nitrogen in nitrite is proved without doubt to be of equal value to plant life as is the nitrogen of nitrate. It is, however, impossible to use soda solution on account of its price, and the use of milk of lime for absorption purposes involves numerous difficulties. It cannot be applied in ordinary absorption towers down which the absorption liquid trickles, as the whole of the spaces in such a tower would very soon be stopped up. We have recently overcome the difficulties attending the absorption with milk of lime, and have succeeded in obtaining a calcium nitrite that has very valuable properties. It is to be manufactured on a larger scale so that already in the present season field experiments may be made as to its manurial properties. This salt is characterized by containing a large percentage of nitrogen—18 per cent., whilst Chili saltpeter only contains 15 per cent. The Norway saltpeter which has been brought into commerce by the Notodden factory contains 13 per cent. of nitrogen. This new air-saltpeter may perhaps be destined to be the nitrogen manure of the future if the field experiments confirm the results hitherto obtained. If, however, it should be necessary to convert the nitrite into nitrate, this can readily be done.

At Notodden, where, as I have already mentioned, the oxidation is effected with Birkeland-Eyde furnaces, the absorption is not effected altogether with alkaline liquids, but water is principally employed. Nitrogen peroxide in contact with water

behaves similarly as with alkalies, only in this case salts are not formed, but the free acids, that is to say a mixture of nitric and nitrous acid. The nitrous acid decomposes into nitric acid and nitric oxide, and this again in contact with air oxidizes to nitrogen peroxide. The absorption with water, is, therefore, a somewhat complicated process and involves continuously a re-formation of nitric oxide, the quantity formed becoming continually smaller

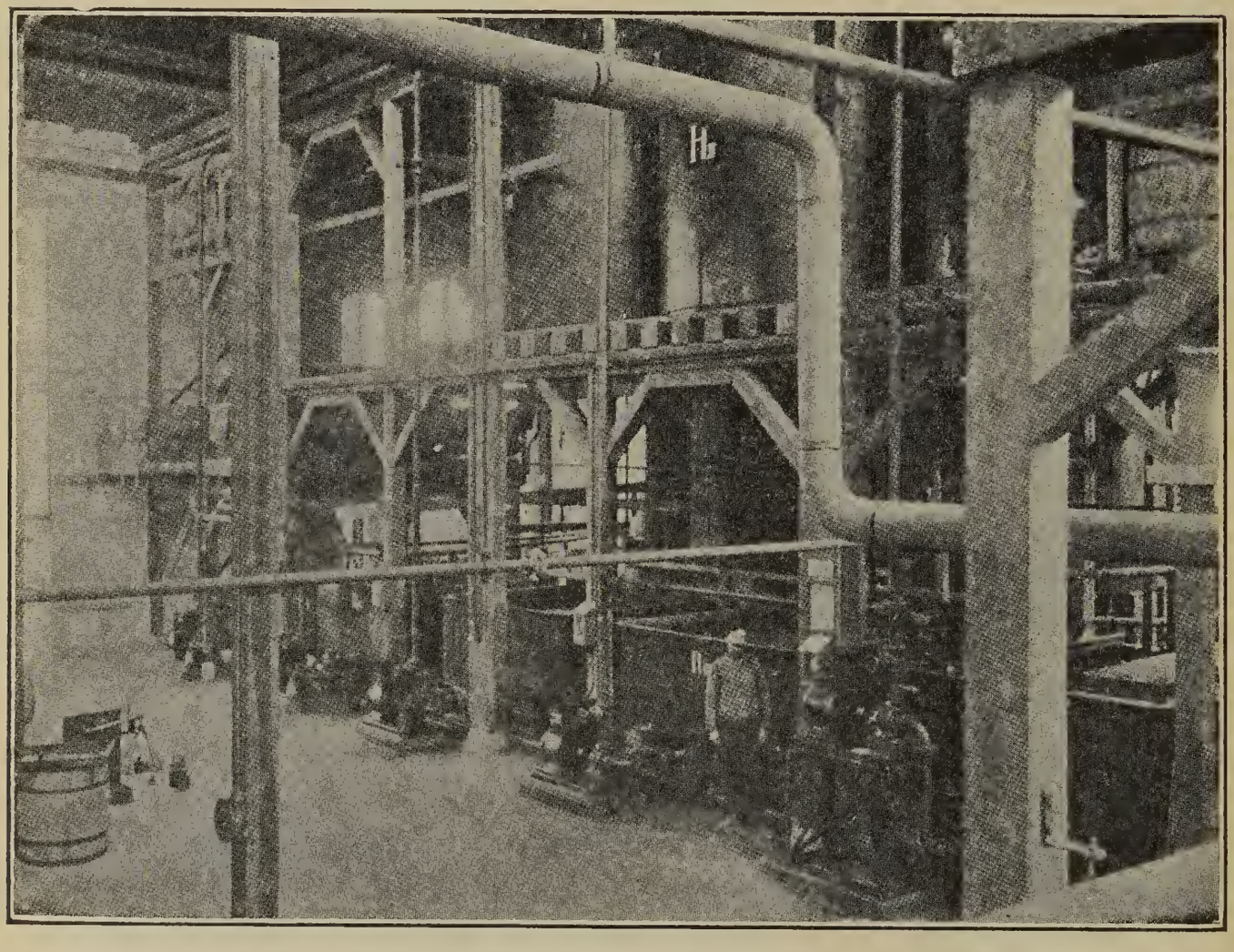


Fig. 10: Absorption Room of the Christianssand Plant.

and smaller. It is consequently impossible to convert the entire quantity of nitric oxide into nitric acid by contact with water, or at least this would only be possible when using absorption vessels of monstrous size; consequently in the factories at Notodden the last portion of the nitric oxide is absorbed with soda, and in this way pure sodium nitrite is obtained, for a complete absorption can be effected with alkaline liquids if only half of the nitric oxide has been converted into nitrogen peroxide. The mixture of NO

and NO_2 behaves as if it were N_2O_3, the anhydride of nitrous acid, and the absorption takes place, nitrite alone being formed. There is a demand for nitrite in the chemical industry, in which it is mainly used for the production of azo dyes.

If desired, the entire quantity of nitric oxide produced in the furnace can be converted into nitrite by working according to the process of our German Patent No. 188,188. We work in this way in our Kristianssand factory. There the nitrite is manufactured which is used in the Ludwigshafen factory of the Badische Anilin & Soda-Fabrik. Sodium nitrite was hitherto obtained by the reduction of Chili saltpeter with lead; this method of production will soon cease to be practiced. It must, however, be borne in mind that the market for nitrite is very limited; its production will never be of any considerable importance for the artificial nitrate industry. It will be possible by the use of about 12,000 h. p. to produce all the nitrite required in the whole world.

When absorbing with water, the more perfect the oxidation of the nitric oxide to nitrogen peroxide is, the more nitric acid will be formed immediately on absorption, and it is therefore advisable to insert between the cooling apparatus and the first absorption apparatus a so-called oxidation vessel in which the gases remain for some time and can consequently become converted as fully as possible into nitrogen peroxide. The problem of absorbing the nitrous gases may at first sight appear a simple one, but besides the difficulties that have already been mentioned, it involves others. The large quantities of air that have to be dealt with make it necessary to use rather large absorption chambers in order to wash the nitrous gases out of the air. The difficulties increase, as might be expected, with the degree of dilution in which the nitric oxide is obtained, and it can readily be understood that of two processes which use equal quantities of energy for the production of the same quantity of nitric oxide, that one will be the more valuable which yields the gases in the most concentrated form.

A large number of suggestions have been made with the object of simplifying the absorption. Absorption with concentrated sulphuric acid can be very readily effected. The product obtained is nitric acid and nitrosyl sulphuric acid, but no method is known of converting the nitrous gases from this sulphuric acid

in a simple way into another compound, so that this method of absorption can only be used where there is direct use for nitrous sulphuric acid. It has been suggested to absorb the nitrous gases from the mixture with air by absorption with bodies which upon subsequent heating give the gases off again in a concentrated form. This method is neither new nor has it any technical advantages. Another suggestion is to cool the air containing the nitrous gases so as to liquefy or even solidify the nitrogen dioxide. For this purpose a complicated apparatus is necessary, and it involves compression of the gases, so that in my opinion it is not suitable for manufacture on the large scale.

It is, however, to be anticipated that there will be many improvements made on this problem of absorption. The process that Schlœsing has patented is of interest. The nitrous gases are absorbed while still hot with quicklime in the form of briquets. At first calcium nitrite is formed, but this, under the continuous action of air and nitrous gases, is converted into nitrate, so that the final product directly obtained is calcium nitrate in the solid state.

By bringing the nitrous gases into contact with water, as is done at Notodden, a dilute nitric acid results. It cannot readily be obtained of a higher strength than 30—40 per cent. by direct absorption. In order to prepare concentrated nitric acid from this, any of the known ways can be used, for instance, by distillation the dilute nitric acid can be converted into 60 per cent. nitric acid. Upon repeating the distillation, adding some agent to combine the water, for instance concentrated sulphuric acid, or dry calcium nitrate, this can be converted into a concentrated nitric acid. The Saltpetersäure-Industrie-Gesellschaft has taken out a patent for a process for concentrating nitric acid electrolytically, but I cannot think that this process will work more cheaply than the other process. In the literature there is a statement to be found that by mixing water vapor with the nitrous gases the condensation to nitric acid can be simplified, indeed it is stated that in this way fairly concentrated nitric acid can be obtained; this is not the case.

The direct conversion of the nitrous gases into nitric acid without the intermediate re-formation of nitric oxide, which is the cause of the slow procedure of the absorption, can be effected by

means of ozone. With the aid of ozone, indeed the anhydride N_2O_5 could be produced. A cheap method of producing ozone might essentially simplify the absorption.

As a rule, when it is desired to produce nitrate, the dilute nitric acid is simply poured without any further treatment on to limestone, calcium carbonate. In this way a solution of calcium nitrate is formed. This is the method of procedure at Notodden. This dilute solution of calcium nitrate is evaporated down in a vacuum apparatus by the aid of steam produced with the hot gases that leave the electric furnaces. No fuel has been used at all.

When the solution has been sufficiently concentrated it is allowed to solidify, and the crystalline cakes obtained are ground to powder. The lime-saltpeter thus obtained is packed in wooden barrels containing 100 kg. (about 2 cwt.). Packed in this way it comes into commerce in a uniform quality containing 13 per cent. of nitrogen, and has already been known for a few years as Norway saltpeter.

A factory four times as large as that in Notodden is now being erected on the Rjukan. The Badische Anlin & Soda-Fabrik and the allied firms, Farbenfabriken vorm. Friedr. Bayer & Co., Elberfeld, and Actiengesellschaft für Anilinfabrikation, Berlin, have combined with the Norwegian Hydroelektrische Stickstoffgesellschaft, to which the Notodden factory and the Birkeland-Eyde patents belong, and have joined them in harnessing Norwegian waterpowers. With reference to this union, it has recently been arranged that the work of granting licenses for both processes should be placed exclusively in the hands of the Badische Anilin & Soda-Fabrik.

The Rjukan factory will be the first factory in common. The harnessing of the Rjukan water power is in a fairly advanced condition. This power is situated in the interior of Telemarken and has a total fall of about 560 meters with a regulated flow of 47 cbm. per second. This will yield in round figures 250,000 el. h. p. The harnessing is being effected in two steps of 280 meters fall each. For the first fall 10 turbines of 14,000 h. p. each will be erected, and this plant with the corresponding nitrate factory is to start work in the year 1910. A normal gauge railway has been newly erected for a distance of 46 km, and a ferry connection instituted along the Tin See, which is 40 km in length.

In this way nitrate will be brought to Notodden, then by lighters to Skien. Here large warehouses will be built and the further transport with ocean steamers will be organized from this port.

Besides this power, the Badische Anilin & Soda-Fabrik have secured in western Norway the water powers of the Matre and Tyin, and have obtained the requisite concessions. It is proposed to manufacture nitrate with these also. When these projects have all been executed, over 400,000 h. p. will have been made available for the new industry in Norway. But the Badische Anilin & Soda-Fabrik have also the intention of carrying out their process in Germany. Application has been made for the utilization of a south German water power on the River Alz. It is proposed to conduct the water of the Alz into the Salzach, whereby about 50,000 el. h. p. could be obtained. The final decision of the Bavarian Government has not yet been issued.

It can be seen, therefore, that the new industry will utilize enormous water powers, but it is altogether unjustified that the charge of wasting energy is brought against it.

The new industry requires a very cheap power, but apart from this hardly any other requirements need to be fulfilled. The industry can flourish where no other could exist, and is, therefore, well calculated to perform the part of a pioneer in the service of industry and to open up far-off districts to industrial pursuits. At the present time, as a matter of fact, only a very small proportion of that part of the sun's energy which raises water from the sea and deposits it on the mountains, whence it runs in countless streams back to the sea, is used for producing power. The reason is that up to the present there was no industry for which cheap power was the sole essential, and which at the same time could use enormous powers. In most industries the position, the site of manufacture, the supply of raw materials, or factors of that sort are far more important, and for many decades or even centuries most industries will be dependent upon coal.

It is almost superfluous to mention that the new industry is not restricted to the manufacture of calcium nitrate, for the dilute nitric acid obtained by the absorption of the furnace gases can be used for the production of other nitrates which have otherwise been obtained from Chili saltpeter. It is probable that the old methods of producing barium nitrate, ammonium nitrate, and

perhaps even potassium nitrate will soon be superseded by the new process which consists of neutralizing bases of carbonates with nitric acid obtained from the air. Nitric acid will also be employed for splitting up insoluble compounds, in particular minerals such as phosphates, borates, and silicates, giving rise to the production of valuable nitrates, whereas up to the present the cheaper acids, sulphuric acid and hydrochloric acid, have been employed, and the comparatively valueless chlorides and sulphates were obtained. We have already seen that the production of concentrated nitric acid from the dilute acid obtained presents no great difficulties, so that the explosives industry, which is the greatest consumer of nitric acid, will also benefit by its production from the air. It is true that the nitrogen contained in nitric acid has a higher marketable value than when in the form of nitrate, but a careful calculator will not base his calculations on the production of nitric acid, but will have to consider the manufacture of saltpeter, and only when this turns out to be profitable has the industry a prospect of survival.

In this new nitrate industry for the first time a great industry has thus been established for which cheap power is the principal factor, and it is an industry which at the same time can utilize practically unlimited quantities of energy, for the product which is obtained is certain of rapid consumption, which is constantly increasing. This utilization of large quantities of energy has been represented as a disadvantage of the new industry by several critics. It is incomprehensible to me how this can be done, for the success of every industry, the relation between the cost of production and the selling price, is the final deciding point, but it is altogether a matter of indifference what are the items which constitute the cost of production. Indeed it might almost be stated that the position of the new industry is particularly strong, because the principal expense is the cost of the water power, for this will be obtainable in future decades also at the same price as now, whereas wages, the price of coal and other raw materials which are of far greater importance in other industries are constantly increasing. The new nitrate industry will present technical electricians with a series of new problems, and will suggest a large number of inventions. At the present moment we are but at the beginning, but at a very promising beginning, and

when the history of the epoch of the "white coal" comes to be written, the first pages will have to be devoted to a description of the artificial nitrate industry.

DISCUSSION.

MR. EDWARD R. TAYLOR: I had the great pleasure of hearing a similar paper read before the International Congress of Applied Chemistry, and the illustrations herein contained were carried out in the form of demonstrations before us. I must say it was one of the most interesting papers I ever had the pleasure of hearing, and it received the attention of a crowded room upon its presentation.

It seems to me there were only a comparatively few people at the congress who were able to hear that valuable paper, and if there is an opportunity some time here in America for these same demonstrations to be made, we should by all means arrange it. The passage of the electric current through the tube was a magnificent sight. It was arranged within a glass tube, and when the electricity was put on you could see the flame starting at the bottom and going quietly through the tube to the top. It kept passing through as a burning flame during the experiment. The nitrogen was oxidized, and you could see the changes to a brown color in the receiving chamber as the reaction went on. It was exceedingly interesting, and one of the most valuable papers heard at the congress.

A paper read at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 28, 1909. President L. H. Baekeland in the Chair.

PREPARATION AND PROPERTIES OF PURE BORON.

By E. Weintraub, Ph. D.

Introduction.

Boron belongs at present, together with a number of other elements, such as zirconium, uranium, etc., to the class of practically unknown elements.

On account of its great affinity for most other elements, its isolation presents such difficulties that serious attempts to accomplish it are not numerous.

The older chemists, Berzelius, Woehler, etc., found that boric anhydride, borax and other compounds, for instance double fluorides, could be reduced by means of alkali metals and magnesium, but obtained products which were too impure to be considered even as approximately pure boron.

The properties of these complex substances have been transcribed into most text and reference books as those of the element boron. They need not occupy us here at all.

Moissan, in the year 1895, which is the most recent work on the element, found that of these methods of preparation the reduction of boric anhydride by magnesium was the most suitable. He found that if boric anhydride was in excess of the theoretical amount, the product of the reaction, after being submitted to the necessary chemical treatment, contained only a few per cent. of magnesium as an essential impurity; it gave him on analysis a content of 93 per cent. boron. By fusing this product with fifty times its weight of boric anhydride he succeeded in removing the greater part of the magnesium and obtained a product containing 98 per cent. and, in one experiment, as high as 99 per cent boron. The substance thus obtained was an amorphous powder of light brown color, which, according to Moissan, could not be fused, as it volatilized without fusing.

The physical properties of an element cannot be considered as known so long as it has been prepared only in form of an amorphous powder.

The chemical purity of Moissan's powder could also be considered as not sufficiently established, as only small amounts of the material were prepared and few analyses were carried out.

The position of the element boron in the periodic system of elements, near carbon, would, however, make the knowledge of the properties of the element of great theoretical, and possibly practical, interest.

While engaged in a search for elements suitable as a material for incandescent lamp filaments, I undertook the study of the element boron with the purpose of developing a way of preparing it in pure condition and in fused homogeneous form.

I began by studying the reduction of boric anhydride by magnesium. According to the relative amounts of the substances and to the temperature, products were obtained which contained, combined with boron, either

(*a*) a large percentage of magnesium;
(*b*) a considerable amount of oxygen;
(*c*) magnesium and oxygen.

Under no conditions was I able to obtain pure boron.

The substance under (*b*), which is, or at least has the composition of, a sub-oxide of boron, is in its appearance and properties very much like the substance described by Moissan as the pure element.

The work was then directed toward development of new methods, and finally led to the preparation of the element not only in the state of absolute purity, but also in fused condition.

The properties of the element thus obtained pure for the first time are interesting from a purely theoretical point of view and are also, I believe, capable of practical applications.

This article is limited to a general description of methods and results. A detailed description of the methods and apparatus used, of the numerous experiments performed and incidental results obtained during the years occupied by this investigation would require more space than can be allotted to an article and more time than I can give it at present.

Preparation and Properties.

1. *Reaction Between Boric Anhydride and Magnesium.*

The reaction between boric anhydride and magnesium, which is supposed to give boron, corresponds to the chemical equation:

$$2B_2O_3 + 3Mg = 3MgO.B_2O_3 + 2B.$$

According to this equation, the relative amounts of boric anhydride and magnesium would correspond very nearly to the ratio 2 : 1, and this is the ratio which was used in most of the experiments by the older chemists in their attempts to prepare boron. In reality, however, if this ratio is used the product of the reaction contains a considerable amount of magnesium and is to be considered as a magnesium boride rather than the element boron.

Moissan found that by increasing the amount of boric anhydride so as to have the ratio 3 : 1 by weight, the resulting product contained much less magnesium. The product obtained by Moissan contained, according to his analysis, about 93 per cent. to 94 per cent. boron, 3 per cent. to 4 per cent. magnesium and about 1.5 per cent. of a substance insoluble in acid. The product of the fusion of boric anhydride with magnesium consists or dark colored parts and others that are disseminated in the middle of the mass and are of a very light brown color. According to Moissan, these latter parts are to be carefully separated from the dark colored ones, and it is this brown product that, after being treated with water and acids, analyzed as above.

In repeating the experiments of Moissan, I obtained products which contained 88 per cent. to 90 per cent. boron, 2 per cent. to 4 per cent. magnesium, 0.2 per cent. to 0.3 per cent. iron (introduced by the steel ball mill used for grinding the boric anhydride) and 0.5 per cent. to 1 per cent. boron nitride, the sum of all the elements determined differing from 100 in average by 4 per cent. to 5 per cent.

As stated above, the direct product of the fusion does not have a homogeneous appearance, but consists of a dark product rich in magnesium and a brown product supposedly boron. As the two products are more or less intermixed, it is obviously impossible to achieve a complete separation, and, to the presence of the darker colored product (which is rich in magnesium) in the

brown product, could be attributed the content in magnesium found.

It was necessary to improve the method in such a way as to obtain a *homogeneous* fusion.

The difference in composition and appearance between parts of the same fusion could only be accounted for by assuming differences in temperature. The brown material prevails in the center of the mass where the temperature is highest; the dark product prevails on the outside of the mass where the temperature is lowest.

The brown substance, being apparently the product of the reaction at a higher temperature, two methods were recurred to for the purpose of production of a uniform brown fusion, namely: first, using a higher temperature furnace (an electric furnace, for instance); second, carrying out the reaction *in an ordinary gas furnace* on a large scale so that the temperature *inside of the mass* could attain a high value on account of the heat of the reaction itself.

Both methods led to the expected result. When a large crucible was charged with a kilogram or more of the reacting mixture, the resulting fusion was of a uniform brown color throughout except for a thin layer on the very outside which could easily be removed.

After removal of this outside thin layer and the usual chemical treatment of the mass, the resulting powder was of a somewhat lighter color than the one obtained in following Moissan's descriptions, showing the absence of the dark magnesium boride, and analysis showed only 0.11 per cent. magnesium instead of 3 per cent. to 4 per cent., found when the reaction is carried out in the old way.

The preparation of a product practically free from magnesium has thus been achieved.

I expected to find pure boron, but the analysis of the product gave only 83.2 per cent. boron instead of 88 per cent. to 90 per cent. found in the product obtained in the old way. This was a surprising result. The sum of all the elements determined (including boron nitride and traces of iron) was only in the neighborhood of 85 per cent., and 15 per cent. were unaccounted

for. In the product obtained in the old way the sum differed from 100 by 4 per cent. to 5 per cent.

In view of this surprising result, the outer layer of the same fusion, which was dark and contained a good deal of magnesium, was also analyzed. It showed a boron content of 91.77 per cent. and 1.5 per cent. magnesium. In other words, the product rich in magnesium was also richer in boron. It is also significant that the sum of all the elements determined (including boron nitride and iron) was about 94 per cent. instead of 85 per cent., as in the case of the light brown product.

The experiments were repeated a large number of times with analogous results.

Results of the same character, only more uniform, were obtained by the first method, namely, the use of the electric furnace. A vacuum furnace was used to exclude the nitrogen of the atmosphere, which at the high temperatures is very readily absorbed by boron, and, to a large extent, carbon monoxide, which is always present in ordinary electric furnaces and which attacks boron with formation of boron carbide.

The reaction mixture is placed in a graphite crucible; after the reaction the outside layer is removed. The product obtained from the reaction is practically free from carbon if the temperature used is not higher than 1,700°. The reaction product shows only traces of magnesium, 0.6 per cent. boron nitride and 82.5 per cent. boron. The sum of the elements determined differs from 100 by more than 16 per cent.

The question arises what the impurity in the brown magnesium-free product may be. From the method of preparation and the analytical results obtained, this impurity could be one of the following substances:

(1) Water.

(2) Admixed boric anhydride.

(3) Hydrogen introduced during the acid treatment (in which case the substance would be a complex boron hydride).

(4) Oxygen (the substance would then be a boron suboxide).

The assumption (1), namely, the presence of water, was disproven by the fact that heating the powder in vacuum, either in

a carbon furnace or in a quartz tube, to a temperature near 1,000°, did not appreciably alter the analytical results.

The assumption (3), namely, that of the presence of hydrogen, was disproved by a combustion with a mixture of bichromate and carbonate, which failed to show formation of water. This, of course, also helped to disprove assumption (1).

To test assumption (2) the substance was boiled both in water and dilute hydrochloric acid for a number of hours with the result that only very small traces of boric acid could be found in the solution. A test with methyl alcohol also failed to detect the presence of boric anhydride.

There remains, accordingly, only the fourth assumption, namely, the presence of oxygen in the form of a sub-oxide.

The proof of the existence of oxygen in the substance was not easy to obtain. Hydrogen does not reduce the substance, while carbon forms boron carbide only at such high temperatures that experiments of a nature necessary for proof of the formation of carbon monoxide are practically impossible.

The method finally used for the proof of the existence of oxygen in the boron powder was based on the oxidizing action of potassium permanganate. The reducing action of boron on potassium permanganate was noticed by previous experimenters, notably by Moissan, but I found that with the boron obtained in the reaction between boric anhydride and magnesium, this reduction could be carried out in a quantitative way, so much so that a regular titration method could be developed.

A simple reasoning shows that if the impurity in the boron is oxygen in any form the consumption of permanganate would be less than that corresponding to the content in boron, since a part of the boron is in an oxidized condition beforehand. In the case of any other impurity the permanganate titration and boron determination should agree. Calling a the percentage of boron found by analysis, b the percentage of oxygen found by difference,

$$b = 100 - a - Fe - Mg - BN,$$

and neglecting at first the small amounts of iron and magnesium, the consumption of permanganate is found easily to be $a - 11/24 \times b$. In reality, small corrections must be applied

on account of the small amounts of iron and magnesium in the material.

The experiments showed that the titration values of permanganate corresponded very exactly with the values calculated by the above formula; thus in one case the following data were obtained: The analysis of boron gave

$$a = 83.14\%\,;\ Mg = 0.11\%\,;\ Fe = 0.18\%\,;\ BN = 1.5\%,$$

so that b is found by difference to be equal to 15.07 per cent. The above formula gives for the percentage of boron calculated from the permanganate consumption 76.27 per cent. If the consumption of permanganate by iron and magnesium be taken into account the above number becomes 76.34 per cent. The actual titration gave 76.24 per cent.

In those cases where the sum of the analytical results agreed to 100, the permanganate titration agreed with the boron determination. For instance, in one case the analysis showed 88.4 per cent. boron, 8.4 per cent. magnesium, 2.8 per cent. boron nitride, 0.2 per cent. iron, giving a sum of 99.8 per cent. or practically 100 per cent. within the limits of error of the analysis. The permanganate titration gave 90.5 per cent. instead of 90.6 per cent., as calculated from the content of boron, iron and magnesium; a good agreement considering the number of analyses involved.

It is, therefore, established that the brown magnesium-free substance contains oxygen, and I will call it *boron sub-oxide* for the sake of brevity.

The question remains whether the substance is homogeneous in its composition or is a mixture of different compounds. Two different methods were applied to determine this:

The first method consisted in varying the conditions under which the substance was produced and observing whether this changed its composition and properties.

The second one consisted in submitting the substance to different chemical treatments and observing whether any separation of the substance into different parts took place.

The result of the variation of temperature and ratio between boric anhydride and magnesium can be summarized in the three following statements:

1. Low temperature and low ratio of boric anhydride to mag-

nesium favor the formation of boron containing large quantities of combined magnesium.

2. High temperature and a high ratio of boric anhydride to magnesium favor the formation of boron free from magnesium, but containing oxygen in the form of sub-oxide.

3. If the excess of boric anhydride becomes too large it acts as a diluent, and by lowering the reaction temperature leads to products containing both oxygen and magnesium.

Thus in an ordinary gas furnace, when the ratio of boric anhydride to magnesium was below or equal 2 : 1 the product was rich in magnesium; when the ratio was varied between 2.75 : 1 and 5 : 1 the resulting product had approximately the same composition. For instance, the product of the fusion 2.75 : 1 showed 86.37 per cent. boron, 1.1 per cent. magnesium, 0.6 per cent. boron nitride, 0.2 per cent. iron and 11.73 per cent. oxygen. The product 4 : 1 showed 84 per cent. boron, 0.6 per cent. magnesium, 0.2 per cent. iron, only traces of boron nitride and 14.45 per cent. oxygen. With a ratio of 5 : 1 and above, the products began to contain more magnesium (on account of the lowering of the temperature by the excess of boric anhydride) without, however, diminishing their content in oxygen.

Thus by varying the conditions of the reaction in the gas furnace within feasible limits (that is so as to obtain products with only a small percentage of magnesium), the composition of the sub-oxide did not vary very much, although there was a tendency for it to contain a larger percentage of oxygen when the excess of boric acid was larger.

In the electric vacuum furnace, not only the ratio of reacting substances could be varied, but also the external temperature. Variation of the ratio between boric anhydride and magnesium from 3 : 1 to 5 : 1 at a temperature of 1,350°, as well as the variation of temperature from 1,350 to 1,600°, keeping the ratio 3 : 1 constant, caused a variation in the boron content of the resulting product from 84 per cent. to 82.5 per cent., the last number corresponding to the higher ratio and higher temperature.

As is seen in all cases, a product is obtained containing 82 per cent. to 85 per cent. boron and 16 per cent. to 14 per cent. oxygen.

The second method, namely, that of trying to separate the product into different parts, gave similar results.

Partial titration with permanganate was tried. The oxidation was carried out in different stages and the residue analyzed every time. The residue had always approximately the same composition as the original.

Nitric acid of different concentrations was used to bring boron into partial solution. Under certain conditions the residue obtained was a very *light brown color—almost white*—but the content of oxygen in it was only slightly higher than in the original (17 per cent.). Chlorine and hydrochloric gas gave similar results.

Assuming then that a chemical individual is present, the corresponding formula would be $B_{15}O_2$ or perhaps B_7O.

The direct reaction between boric anhydride and magnesium thus having failed to give pure boron, the re-fusion with a large excess of boric anhydride, as recommended by Moissan, was finally tried.

If magnesium boride is re-fused with a large amount of boric anhydride at low temperature, some magnesium is removed, but a few per cent. are obstinately retained. If the temperature is high, the magnesium is completely removed, but oxygen is introduced. Finally if boron sub-oxide is re-fused at high temperature with boric anhydride, it remains practically unchanged.

Simultaneously with the work on reduction of boric anhydride by magnesium a good deal of work was also done on reduction by other metals, such as alkali metals, calcium, aluminium, etc. The results were in general worse than in the case of magnesium, as the affinity of boron for these metals is greater than for magnesium.

The main purpose of the work unudertaken was to prepare pure boron in a fused condition, and I was satisfied from the experiments described above that the reduction of boric anhydride by magnesium (or other metals) is not capable of giving even pure amorphous boron powder. At this point I abandoned any further attempts in this direction.

We will see, however, later, how in taking these impure products as a starting point pure boron can be produced.

2. Reduction of Boron Halides by Hydrogen.

The reduction of chlorides or halides in general by means of alkali metals or metals of alkaline earths has to my knowledge never led directly to the pure element.

In the case of boron, its great affinity for these metals, as shown in particular by the work on reduction of boric anhydride, made the chances of obtaining pure boron by this method very slim. Results obtained by previous investigators did not look very encouraging, and after some work in that direction I concluded that success was not probable.

If it were possible in this reduction to obtain boron free from the reacting metal in some simple way, then passing boron chloride over boron contaminated with magnesium, for instance (obtained by reduction of boric anhydride with magnesium), ought to eliminate the magnesium. Experiments showed that this could not be accomplished.

The reduction of the halides of boron by hydrogen has not been seriously attempted. In the literature I could find only one statement, by Dumas,[1] which I reproduce. In speaking of boron chloride, he says: "But its most precious property so far is to give birth to a solid hydrate susceptible of reduction by hydrogen at the heat of an alcohol lamp. It transforms it to hydrochloric acid and boron, of which one can produce large amounts."

Soon after, Liebig[2] repeated the experiment and obtained only traces of a brown substance and expressed himself as being anxious to find how Dumas had obtained his result.

The statement of Dumas looks on the face of it too optimistic, and from the coursory way in which it was made was apparently not intended to be taken very seriously.

I repeated the experiment. On adding water to boron chloride a white substance is formed which is mostly boric acid, but seems to contain some boron-oxychloride. When heated in a stream of hydrogen a very small amount of a black substance is formed, too small to be analyzed.

Boron oxychloride is not a good starting point on account of its content of oxygen (compare boron sub-oxide) and it was possible that the reduction of boron chloride by hydrogen, if car-

[1] Ann. de Ch. et de Physi., 1825 [V] **31**, 436.
[2] Schweiggers' Journal, 1826 [V] **47**, 126.

ried out at a sufficiently high temperature, would lead to the element.

In the first experiment boron chloride and an excess of hydrogen were passed through a hot glass tube. After a few hours a small amount of soot formed, covering the inside of the tube. It showed the boron flame, but the amount was so small that nothing could be done with it.

To obtain higher temperatures a silica tube, heated by a platinum resistance heater or a blast flame, was used. A small amount of a gray black substance was obtained, which on analysis showed presence of silicon in considerable quantities. This was probably due to the action of the boron formed on the walls of the tube.

This action of boron on oxides is, as I found later, general, beginning with carbon monoxide, which is reduced at red heat, and ending with aluminium oxide, thorium oxide and magnesium oxide, which are reduced at very high temperatures.

In the case of magnesium oxide, this is especially remarkable. We saw that below 1,600° boric anhydride is reduced by magnesium. At very high temperatures (around 2,500°) magnesium oxide, however, is reduced by boron.

It seemed as if this method also would have to be abandoned, when it occurred to me that the high temperature obviously necessary could be obtained by *passing an arc discharge through the mixture of boron chloride and hydrogen.* The boron produced would then come in contact with no walls, except, of course, to a certain extent with the electrodes.

This method proved to be the solution of the problem.

The substances, boron chloride and hydrogen, are both gaseous at room temperature. The reaction products consist of one gaseous substance, hydrochloric gas, and one solid product, namely, the desired element boron. The separation of this product would therefore offer no difficulties whatever.

The experiment soon revealed other advantages of the method. The blast of the arc removes the produced solid particles of boron out of the zone of the reaction by throwing them out on to the cold walls of the vessel or the cold electrodes. The reaction between boron chloride and hydrogen is, indeed, a reversible

one, as is shown by the fact that boron chloride can be produced by passing hydrochloric gas over boron heated to red heat. To insure a good yield in the reduction of boron chloride by hydrogen it is, therefore, desirable to remove the boron as rapidly as possible from the very hot zone of the arc, where the reduction takes place. The mechanical blast of the arc accomplishes this automatically. In this the reaction between boron chloride and hydrogen has the advantage over the reaction leading to nitric acid from nitrogen and oxygen.

A serious difficulty, however, presented itself, which does not exist in the case of the nitric acid process, namely, the introduction of solid impurities from the material of the electrodes into the solid product of the reaction.[3]

I soon found that a direct current arc presented that difficulty to a large degree on account of the unavoidable disintegration of the negative electrode.

An alternating current arc had to be recurred to and then necessarily a high potential arc. Even in this case, however, most of the electrodes introduced impurities into the product, both on account of a slight disintegration and on account of their affinity for boron. With graphite electrodes, for instance, the product obtained contained as much as 2 per cent. of carbon.

The difficulty was finally overcome by the use of copper electrodes. Under the conditions of the experiment not the slightest disintegration took place, and the affinity of copper for boron is so slight that boron could be directly fused on the electrode without being contaminated with copper.

The method finally developed consisted accordingly in running one or more alternating current arcs, fed by a high potential transformer, between water-cooled or air-cooled copper electrodes, in a mixture of boron chloride with a large excess of hydrogen, in a glass or copper vessel.

The boron is partly thrown off in form of fine powder onto the walls of the vessel or onto the electrodes. It partly settles on the ends of the electrodes where it grows in form of small rods. After a while the arc thus runs between two boron electrodes, and if the current of the arc is of proper value the rods

[3] This difficulty would not exist if a sufficient amount of pure boron were at one's disposal to be used as material for the electrodes.

melt down to boron beads which eventually fall off, whereupon the same process repeats itself.

The product obtained consists, therefore, partly of powder and partly of fused lumps. The powder has to be washed with water to remove a small amount of boric anhydride, the formation of which is due to traces of air set free from the electrodes and the walls of the vessel. After the small amount of boric anhydride mixed with the powder is thus removed, the analysis gives 99 per cent. to 99.5 per cent. boron. As neither copper nor hydrogen could be detected in the powder, the missing 0.5 per cent. to 1 per cent. may possibly be due to presence of a small amount of boron sub-oxide. It seems, in fact, impossible, unless very exceptional precautions are taken, to avoid oxygen in the gases and possibly traces of boron oxychloride in the boron chloride used. As the permanganate titration does not go quantitatively with boron produced under these circumstances, the presence of a small amount of boron sub-oxide could not be proved.

The fused lumps of boron analyze to 99.8 per cent.-100.2 per cent. The difference from 100 is due to a trace of silica which is introduced into the boron during the process of pulverization in the agate mortar and to the unavoidable analytical errors.

Both the method of formation and the analysis prove the absolute purity of the fused lumps of boron.

Within certain limits, the yield obtained is the greater the larger the excess of hydrogen used; naturally, however, the amount of boron obtained per unit of time is small. The yield is also greater the larger the surface of contact between the arc and the gas. A number of arcs in the same vessel or in vessels mounted in series is, therefore, advantageously used. The best yield I ever obtained in one vessel was 50 per cent. of the theoretical. As a rule, however, 30 per cent. can be considered satisfactory, and at the same time a reasonable speed of boron formation is obtained.

The boron chloride can be condensed out of the escaping gases and used over again.

The mixing of the gases can be carried out either by passing hydrogen over the surface of boron chloride placed in a vessel surrounded by a refrigerating mixture or by allowing boron

chloride to drop into a vessel, kept a little above room temperature, through which hydrogen is passed.

In the arrangement of the arcing apparatus all the well-known modifications, such as use of a magnetic field, the introduction of gases through the electrodes themselves, etc., can be applied.

The boron powder obtained in the process can be purified and fused down by two different methods developed in the course of this investigation. These methods are very valuable in the treating of refractory materials and have become regular tools in the research laboratories of the General Electric Co. Their purpose is to treat or melt refractory materials under conditions where no contamination with foreign material is possible. The first consists in the use of a mercury arc, either in vacuum or in an indifferent gas and is best applicable to materials having an appreciably high conductivity.

It is based on the evolution of heat which takes place at the anode of a mercury arc (constant voltage drop multiplied by current) and on the fact that most refractory materials, such as tungsten, tantalum, boron, etc., have no affinity whatever for mercury.

The arrangement, which is used in a large number of modifications, can be termed a *mercury arc furnace.*

The second method is based on the use of a high potential arc in an indifferent gas and is especially applicable to the treatment, purification and melting of substances possessing only a very slight conductivity. This method was especially used in this boron investigation and is one of the methods used in Section 4 of this article.

3. *Physical Properties of Pure Boron.*

The melting point and the sublimation point of pure boron lie near together and between 2,000° and 2,500° C.

Under atmospheric pressure, boron readily melts, as we saw in Section 2. In vacuo, boron first volatilizes, more and more rapidly as the temperature is raised, until apparently under the pressure of its own vapors it begins to melt. When once the surface is thoroughly fused the tendency to volatilize is greatly reduced, and then the boron can be fused in vacuo with only a small loss in weight.

The accurate determination of the melting point could probably best be carried out by melting boron in hydrogen by means of a high potential arc, the use of which is mentioned above. I have not attempted this determination and take my estimate, of its being between 2,000° and 2,500° C, from a rough comparison of brilliancy of surfaces.

The considerable vapor tension of boron, which begins to show itself in blackening the walls of the exhausted globe, even at a temperature of 1,600°, makes boron as such useless as a material for incandescent filaments.

In *hardness,* fused boron is inferior only to diamond.

The *fracture* of well fused boron is conchoidal, showing no signs whatever of micro-crystalline structure. In this, boron greatly resembles diamond.

It is inferior to good specimens of black diamond in its *toughness* or strength. To the tremendous pressure under which diamond is formed its characteristic toughness is usually ascribed, and it is probable that if boron were fused under great pressure it would acquire similar characteristics.

For the purpose of artificial production, which has not yet given practical results in the case of diamond, the conditions are favorable in the case of boron on account of its considerably lower melting point.

We come now to the most interesting property of fused boron, namely, its *electrical conductivity.* At ordinary temperature boron is a very poor conductor, but its conductivity rises with the temperature with very exceptional rapidity. Between room temperature (23° C.) and dull red, the conductivity of fused boron increases about 2,000,000 times, a rate of increase unknown in any other element with the possible exception of selenium in the narrow range below the melting point of the latter. Above dull red the conductivity still continues to increase very rapidly, and at bright red heat boron is a relatively good conductor.

A similarly great fall in resistance is exhibited by oxides at temperatures above red heat; boron, however, shows this behavior around room temperature, where quantitative study of the relation between temperature and resistance is possible.

For small intervals of temperature, the resistance drops geo-

metrically for equal temperature differences; in other words, as a first approximation for not too large intervals of temperature, *the resistance is an exponential function of the temperature.*

Around room temperature the interval of temperature for which the resistance drops to half its value is about 17°. This interval slowly increases and is approximately 30° around 300°C. Below is given one set of resistance measurements on a certain piece of boron. This table has no pretentions to being accurate, as very slight errors in the temperature determination cause very great differences in the resistance. For this reason two sets of measurements never completely agreed.

Temperature	Resistance, in Ohms
27°	5,620,000
45	2,590,000
60	1,500,000
70	1,060,000
80	758,000
90	560,000
100	395,000
110	285,000
120	238,000
130	194,000
140	134,000
150	107,000
160	77,000
170	55,000
180	46,000

The measurements at high temperatures are rendered somewhat inaccurate, not only by the factors mentioned above, but also by some thermo-electromotive forces of small magnitude, the origin of which has not yet been cleared up. Thus in a second set of measurements, which agreed fairly well at lower temperature, larger discrepancies appeared at temperatures above 100°. The next table carries the measurements up to 300°, the resistances being simply those of the particular piece of boron tested.

Temperature	Resistance, in Ohms
140°	107,000
160	55,000
170	40,000
180	30,500
190	22,000
210	13,800
220	10,700
250	5,300
200	2,900
290	2,250

Finally the same piece of boron was put into an exhausted globe and the measurement of resistance continued to see how far the resistance would drop. At beginning red (visible in a darkened room) the resistance was only 5 ohms. Thus between 27° C. and beginning dull red heat the resistance of the boron piece dropped from 5,620,000 ohms to 5 ohms. The values at the lower temperature are certainly too low on account of the heat developed by the small current (0.000001 amp.). This is especially serious on account of the high resistance of boron, which makes the energy dissipated high even at very small currents, and on account of the small specific heat of boron.

The determination of the specific resistance (resistivity) of boron from these measurements is somewhat difficult on account of the irregular shape of the lumps used. At 23° C. the resistivity has the approximate value of 1.7×10^6, or about 10^{12} times that of copper.

Towards moderate applied voltages boron behaves, however, as a relatively good conductor, on account of the change in resistance which takes place automatically and rapidly, due to the heating of the boron by the current passing through it.

A substance with such marvelous electrical properties is naturally capable of a number of technical applications, of which I mention the following:

A motor-starting device.

A sensitive pyrometer and bolometer.

A wireless detector.

A compensator for positive resistance changes due to temperature, etc.

The hardness of boron, combined with its conchoidal fracture, make it applicable to use as jewels, dies, etc.

All these technical applications are now in process of development.

4. Methods of Preparation of Pure Boron Based on Dissociation.

The preparation of very large amounts of boron by the reduction of boron chloride with hydrogen in an arc is somewhat laborious if carried out on a laboratory scale.

As a result of an investigation of a number of boron compounds, methods based on the dissociation of some of them at

high temperature were developed. These methods can only be touched upon in this article, a full description of them being reserved for another time.

Three different compounds of boron were found up to the present which are capable of dissociating at high temperature, leaving pure boron.

1. *Magnesium boride* (obtained in the reaction between boric anhydride and magnesium when the first is not in excess) and similar compounds. The dissociation begins at relatively low temperature (1,200°), especially in vacuo, and is quite rapid at 1,500°.

2. *Boron sub-oxide* (obtained in the reaction between boric anhydride and magnesium when the first is used in excess and a high temperature applied). The dissociation begins only at high temperature and is accompanied by volatilization of a part of boron sub-oxide (especially if it is carried out in vacuo).

3. *Boron nitride.* The dissociation of this compound only takes place at a very high temperature.

If the dissociation of these compounds is carried out in ordinary electric furnaces where the heating element is carbon, the product obtained is contaminated somewhat with carbon.

5. *Preparation and Properties of Solid Solutions of Carbon and Other Elements in Boron; Chemical Metallization of Carbon.*

After pure boron in fused form had been prepared and its properties ascertained, an investigation was started on the influence of small amounts of other elements dissolved in boron. Carbon, silicon, magnesium, aluminium, etc., were tried.

I will describe here only the properties of solid solutions of carbon in boron, as being perhaps the most remarkable of all.

If carbon is introduced into boron in small amounts the cold conductivity is enormously raised and the negative temperature coefficient of resistance reduced. The effect is so great that it is not an easy matter to get accurate results. The magnitude will be shown if I state that a few tenths of 1 per cent. of carbon reduce the cold resistance of boron in the ratio of 12: 1.

Between pure boron and boron containing about 8 per cent.

carbon there is a most interesting region in which almost any desired negative temperature coefficient of resistance can be obtained.

The resistivity goes down as the carbon content increases, which may be very advantageous for many practical purposes.

Above 8 per cent. carbon the temperature coefficient is of a magnitude which can be found readily in other substances.

The solid solutions of small amounts of boron in carbon exhibit still more remarkable properties.

All the known forms of carbon which conduct at all, whether amorphous carbon or graphite or carbon made by carbonizing cellulose (base of a carbon filament in incandescent lamps) or deposited from volatile hydro-carbons, have a negative temperature coefficient of resistance.

The only exception is formed by the so-called "metallized" carbon produced by the General Electric Co. by heating carbon deposited from gasoline to a very high temperature in an appropriate electric furnace.

This species of carbon has relatively low resistivity and a positive temperature coefficient of resistance.

The same change in the properties of the carbon is produced, as I find, by incorporating into it a very small amount of boron, an amount so small that it can be detected only by flame reaction, but cannot be determined analytically; in other words, *boron has a "catalytic" effect, transforming metalloidal carbon into metallized carbon.*

This effect must not be confused with the well-known graphitizing action which a large number of elements have on carbon. In the case of graphitization a material is produced which decreases in resistance with increase of temperature.

The metallizing action of traces of boron is specific to this element. All other elements tried, particularly iron and silicon, do not produce that result.

Whatever the cause of the peculiar action of boron, it is established that by its means the *chemical metallization* of carbon is accomplished for the first time.

It is remarkable that a substance like boron, with an enormous negative temperature coefficient of resistance, when dissolved

in traces in carbon, which also has a negative temperature coefficient of resistance, transforms the latter into a positive one.

Summary.

1. The element boron has been prepared for the first time in pure fused form.

2. A method of carrying out reactions between volatile substances leading to a solid product has been developed.

3. The properties of solid solutions of different elements in boron have been investigated.

4. The chemical metallization of carbon has been realized.

5. The existence of a boron sub-oxide has been established.

The above paper is a contribution from the Research Laboratory of the General Electric Company, at West Lynn, Mass., and the experimental work was carried out with the assistance of E. Kraus and G. Weintraub.

DISCUSSION.

Mr. Carl Hering: I did not hear Dr. Weintraub say how the material is handled after it is in the form of a metal rod, whether it can be drawn into wire or welded, or how to handle it and get it into desired shape.

Mr. Weintraub: Boron powder is compressed into a thin rod and sintered by current, and then heated in an atmosphere of boron chloride and hydrogen. It cannot be drawn any more than diamond can. After the boron is compressed, you can deposit more and more of the boron, and in that way you can get any shape, a rod, a plate, or anything you desire.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909, President L. H. Baekeland in the Chair.

THE PREPARATION OF CALCIUM.

By Francis C. Frary and Walter L. Badger.

Though discovered in 1808 by Davy,[1] and apparently at about the same time by Berzelius and Pontin,[2] the metal calcium has, until recent years, been a chemical curiosity of more value on a museum shelf than in the chemist's laboratory. As it is now being produced on a commercial scale at Bitterfield, Germany, it has seemed worth while to study its preparation with a view to ascertaining under what conditions it could conveniently be prepared in the laboratory.

The preparation of calcium by the reduction of its chloride with sodium has been investigated by Lies-Bodart and Jobin,[3] Caron,[4] and Moissan.[5] The latter was probably the first to prepare the pure metal in large quantities and to study thoroughly its properties and behavior. His method of re-crystallizing the metal from solution in sodium, though tedious, was the best for preparing the pure metal, until the difficulties surrounding the electrolysis of molten calcium chloride had been overcome, and is probably the final word of the purely chemical process for its production.

The production of calcium-amalgam and its subsequent distillation to obtain the free metal have been studied by Davy,[6] Berzelius and Pontin,[7] Hare,[8] and Bunsen.[9] This seems to be the only way in which the metal can be prepared from aqueous solutions of its salts.

[1] Phil. Trans., 1808, 343, 354; Works of Sir Humphrey Davy, **5**, 102; Ann. Chim. Phys., 1809, **70**, 189; 1810, **75**, 129.

[2] Letter to Davy, quoted by him, *loc. cit.*, also Gilbert's Ann., 1810, **36**, 247.

[3] Compt. Rend., **47**, 23; Chem. Gaz., 1858, 329; Repert. Chim., **1**, 15; Ann., **108**, 20; Ann. Chim. Phys. (3), **54**, 363.

[4] Compt. Rend., **48**, 440; Ann., **111**, 114; Chem. Gaz., 1859, 165.

[5] Compt. Rend., 1898, **126**, 1753; J. Chem. Soc., 1898, **74**, ii, 578; Ann. Chim. Phys., 1899, (7), **18**, 289.

[6] *Loc. cit.*

[7] *Loc. cit.*

[8] J. Pr. Chem., 1840, **19**, 249.

[9] Ann., 1854, **92**, 251.

Bunsen seems to have been unsuccessful in electrolyzing molten calcium chloride, but two years later Matthiessen,[10] in his laboratory, succeeded in isolating what was probably a rather impure metal by the electrolysis of a mixture of the chlorides of calcium and strontium. Frey[11] used the same method.

In 1898, von Lengyel[12] obtained metal of 99.2 per cent. purity by the electrolysis of calcium chloride with the use of a porous earthenware diaphragm. Only small quantities were obtained, and the porous diaphragm was evidently the cause of the high voltage required (70 to 110 volts for 10 to 18 amperes), and undoubtedly caused rapid contamination of the bath and the metal.

In 1902, both Borchers and Stockem,[13] Ruff and Plato,[14] and Arndt,[15] prepared the metal on a larger scale and with better apparatus. The principal point made by Ruff and Plato was the use of a mixture of 100 parts of the chloride and 16.5 parts of the fluoride as the electrolyte, thus reducing its melting point to 650°. Borchers and Stockem laid special stress on their apparatus, in which the metal was deposited in the form of a sponge in the bottom of the crucible, and, after expelling the excess of electrolyte by squeezing with a pair of tongs, was removed and remelted, as they ingenuously remark, "in an atmosphere free from oxygen." As the metal combines, not only with oxygen but also with all other gases except the helium group (Moissan, *loc. cit.*), it is hard to believe that this part of the process was actually performed in the laboratory. They believed that if molten calcium was left in contact with its chloride a subchloride, CaCl, would be very rapidly formed. But Guntz and Basset[16] showed that no subchloride, but only a mixture of the oxide and hydride was formed, so it seems that deposition of the metal in a

[10] J. Chem. Soc., 1856, 8, 27; Ann., 93, 277; Ann. Chim. Phys., (3), 44, 60.

[11] Ann., 1876, 183, 367.

[12] Math. naturw. Ber. Ungarn, 14, 180; Chem. Centr., 1898, ii, 262; J. Chem. Soc., 1899, 76, ii, 218.

[13] Z. Elektrochem., 1902, 8, 757, 938; J. Chem. Soc., 1903, 84, ii, 19, 145, 211; Ber., 1903, 36, 17; Chem. Centr., 1902, i, 1090; 1903, i, 276, 619, ii, 751; D. R. P. 144,607, U. S. Pat. 808,066.

[14] Ber., 1902, 35,, 3612; 1903, 36, 491; J. Chem. Soc., 1903, 84, ii, 19, 211; 1904, 86, ii, 731; Chem. Centr., 1903, i, 7, 497; 1904, ii, 802; D. R. P. 153,731; U. S. Pat. 806,006.

[15] Z. Elektrochem., 1902, 8, 861; J. Chem. Soc., 1903, 84, ii, 76; Chem. Centr., 1903, i, 8.

[16] Bull. Soc. Chim. Paris, 1906, (3), 35, 404.

spongy form would have few advantages, if it must be remelted later.

In fact, Goodwin,[17] and von Kugelchen,[18] working with furnaces essentially similar to that of Borchers and Stockem, obtained better results by keeping the temperature high enough so that the molten metal was disengaged from the cathode and rose to the top of the bath, where it was removed with a ladle.

In 1904 a great advance was made, when the method of drawing out the calcium as a rod attached to the cathode was described.[19] This seems to be the key to success, and to give much better results than the other methods, as we find Goodwin,[20] a year later, describing a similar arrangement as much preferable to his earlier furnace. In the same year, Woehler[21] describes the electrolysis of calcium chloride in an iron crucible, using an iron rod as a cathode and a carbon anode. He used the electrolyte recommended by Ruff and Plato (*loc. cit.*) and draws out a solid stick of calcium. The iron crucible could scarcely be very satisfactory.

In 1904, Moissan[22] showed that if a carbon cathode be used, both calcium and carbide are obtained.

In 1906, Tucker and Whitney[23] improved the apparatus of Goodwin by water-cooling the cathode and leaving a thin bottom of graphite between the electrolyte and the cooling coil. Fluorspar was placed on the bottom of the crucible and molten calcium chloride poured over it. A current efficiency of 60 per cent. was obtained.

In 1908 Price and Horry[24] patented the use of an anode of calcium carbide, and in 1909 Cowper-Coles[25] patented the use of a disk cathode which revolves and removes the metal from the electrolyte.

Since the method of Tucker and Whitney appeared to be the

[17] J. Am. Chem. Soc., 1903, **25**, 873.

[18] Z. Elektrochem., **14**, 143; Chem. Centr., 1908, ii, 1602; U. S. Pat. 864,928.

[19] Elektrochemische Werke, Berlin, D. R. P. 155,433; U. S. Pat. 813,532; Chem. Centr., 1904, ii, 1180; Rathenau, Z. Elektrochem., **10**, 508; Chem. Centr., 1904, ii, 759.

[20] J. Am. Chem. Soc., 1905, **27**, 1403.

[21] Z. Elektrochem., 1905, **11**, 612; J. Chem. Soc., 1905, **88**, ii, 708; Chem. Centr., 1905, ii, 1012.

[22] Compt. Rend., 1904, **138**, 1194; J. Chem. Soc., 1904, **86**, ii, 483.

[23] J. Am. Chem. Soc., 1906, **28**, 84.

[24] U. S. Pat. 886,857.

[25] Brit. Pat. 24,396; C. A. 1909, **3**, 1498.

most promising, it was chosen as a starting point. We also attempted some work with the Borchers type of furnace, but found that the maximum current available (250 amperes) would not keep the melt fluid, so were unable to directly compare the two methods, as we had hoped to do. As the result of our work, we found the following difficulties with the apparatus of Tucker and Whitney: (1) Poor contact between the crucible and the copper strip clamped around it; (2) rapid destruction of the crucible, due to oxidation and softening of the graphite so that it fell off as a powder; (3) the anode effect.

To remedy the first, we plated a layer of copper 1/16 to 3/32 inch in thickness upon the lower part of the crucible. This made a good electrical contact, and also increased the efficiency of the water-cooling of the bottom, as the cooling coil made better contact with the metal than with the powdery graphite surface. The water-cooling also prevented destructive oxidation of the copper. To preserve the crucible, we tried to give the upper part of it a coating of carborundum fire-sand and water-glass, but found that this always cracked off when the crucible became hot. A form was then made from an empty can and fitted about the upper part of the crucible so as to leave an annular space around it, allowing the tamping in place of a mixture of carborundum fire-sand, fire-clay and barium sulphate, moistened with water-glass, the whole forming a layer ½ inch thick around the crucible and not only protecting it from oxidation, but diminishing the trouble due to the anode effect.

This "anode effect" is a peculiar and interesting phenomenon, apparently first mentioned by Bunsen (*loc. cit.*) who complains of the interruption of the current soon after starting the electrolysis, caused, as he thought, by the formation of a layer of either silicon or lime on one of the electrodes. Similar effects have been observed by Lorenz,[26] Borchers,[27] Muthmann, Hofer and Weiss,[28] Goodwin,[29] Woehler,[30] and Thompson,[31] and investigated and discussed by Willner.[32] The "effect"

[26] Z. anorg. Chem., 1899, **19**, 246; "Die Elektrolyse geschmolzener Salze," **3**, 44.
[27] "Electrische Öfen," 1907 edition, pp. 13 and 14.
[28] Ann. 1902, **320**, 238, 249.
[29] J. Am. Chem. Soc., 1903, **25**, 873.
[30] Z. Elektrochem., 1905, **11**, 612.
[31] Electrochem. Met. Ind., 1909, **7**, 19.
[32] Dissertation, "Studien über die Schmelzflusselektrolyse der Erdalkalichloride," Berlin, 1907.

manifested itself in our experiments as follows: A sudden hissing noise would be observed, the amperage would drop from about 100 to 50, while the voltage rose from 18 to 60 or 70. The disturbance would last from one to perhaps twenty seconds, and then disappear as suddenly as it came. While it lasted, the current seemed to pass from the crucible (anode) to the electrolyte by means of a series of small arcs, apparently through a gaseous layer, and the electrolyte did not seem to "wet" the crucible. In Fig. 1 these variations are shown graphically from readings made

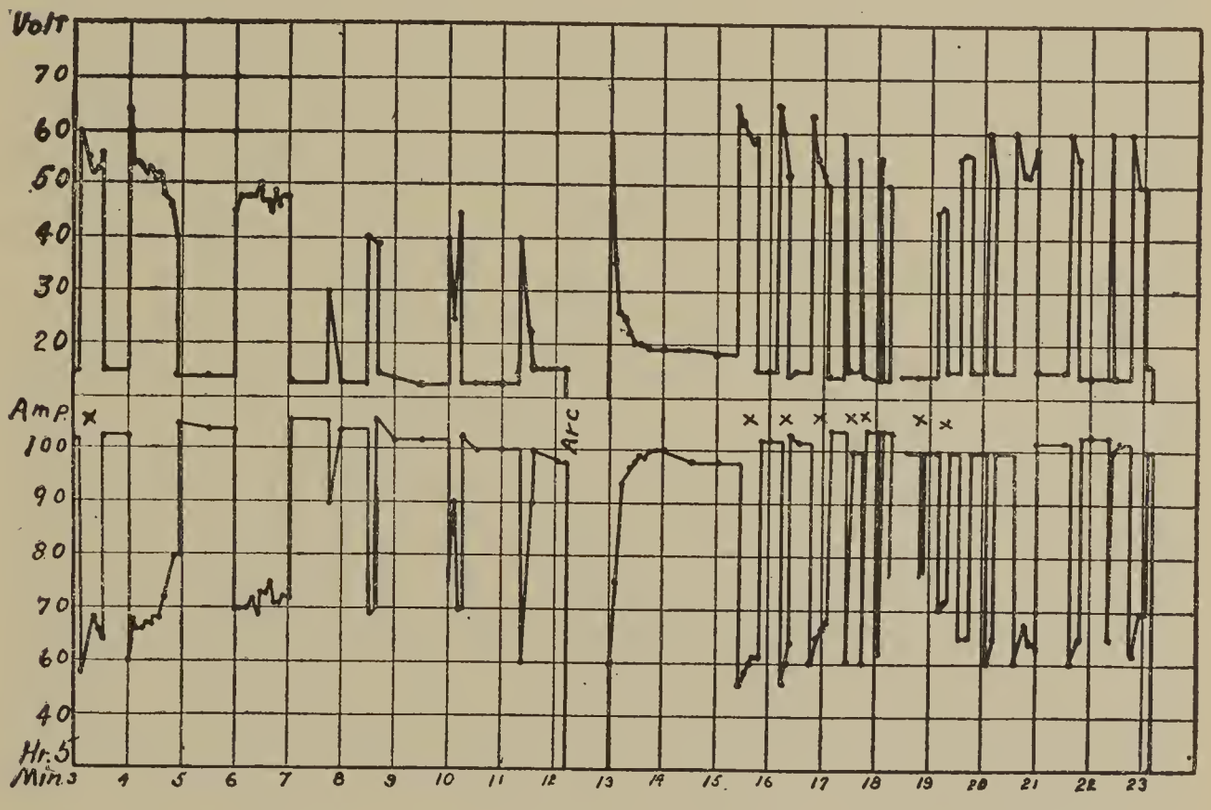


FIG. 1.

during one of the runs with an unprotected crucible. In one of the earlier runs, the effect occurred with remarkable regularity, taking place at intervals of almost exactly a minute, for more than fifteen minutes. It was noticed that the effect seldom occurred during the first few runs in a new crucible, but after the crucible had been used for a while the effect would appear.

There have been several theories to account for this phenomenon. In the electrolysis of cryolite, it seems to be due to an impoverishment of the bath and is remedied by adding more alumina.[33] It has been described as a variety of the spheroidal

[33] Seligman, Electrochem. Met Ind., 1909, **7**, 148.

state, which it certainly resembles in appearance. Borchers ascribes it to excessive current density at the anode, while Willner believes it is due to a layer of silica which he considers would be deposited at the anode if the melt contained a silicate in solution. As most of his experiments were conducted in porcelain crucibles, the source of the silica is easily found. In experiments made in an iron box protected by a layer of the solidified electrolyte, he had much less trouble with the effect, especially if the anode was frequently changed for a fresh one. In a magnesia crucible, containing only 0.08 per cent. silica, the effect would appear only about every five minutes. He rarely had any trouble with the electrolysis of calcium chloride, but with strontium, and especially with barium chloride, the effect was very troublesome. This he ascribed to the smaller solubility of the silica in the latter two salts. In the electrolysis of calcium chloride, he was able to cause the effect to take place by the addition of a pinch of silica.

On the contrary, with the apparatus of Tucker and Whitney, in several electrolyses of strontium and barium chlorides, each of several hours' duration, we had never observed the phenomenon, while in the same apparatus we sometimes had much trouble in the electrolysis of calcium chloride. The chloride used was Merck's C. P. anhydrous: analysis of a 6 gm. sample showed the presence of no weighable amount of silica; 0.002 per cent. should easily have been detected if present. The anode was always Acheson graphite, and as the inner surface was only very slowly and slightly attacked it seems improbable that the pound or more of the electrolyte could have been seriously contaminated by the traces of silica contained in it, especially as calcium was continually being produced and fresh chloride added. Since various persons have shown that silica is readily reduced by metallic calcium, and up to 0.84 per cent. of silicon has been found in commercial metallic calcium[34] and Goodwin (*loc. cit.*) found that his calcium on remelting contained 0.77 per cent. of it, it seems hardly possible that it would accumulate to any extent in the electrolyte. A sample of our poorest electrolyte showed only 0.10 per cent. of silica; this electrolyte had been used in the Tucker and Whitney furnace, poured out and used in the Borchers fur-

[34] Setlik, Z. Elektrochem., 1906, **12**, 390; Chem. Ztg., 1905, **29**, 218; Chem. Centr., 1905, i, 994.

nace, chiseled out of this, swept up and used again, once more chisled out and swept up, and finally used in the Tucker and Whitney furnace again. As several hundred grams of ordinary powdered fluorspar had been used on the bottom of the Borchers furnace, it is not strange that some silica should be found. But when this electrolyte was used in the Tucker and Whitney apparatus no anode effect was found.

All our experience points to the anodic current density as being the important factor in the production of this effect. It is quite possible that the presence of excessive amounts of silica might be a contributing cause: the formation of a film of silica over part of the anode raising the current density on the rest above the critical point. The detailed explanation of the phenomenon seems to us to be as follows:

Under any particular set of circumstances, for any given electrolyte, there is a critical anodic current density, above which the gas will be evolved fast enough so that its bubbles unite into a sheet of gas, through which the current must pass by means of arcs. This, of course, by reducing the current, reduces the current density; but the power consumed is nearly doubled, and all of the increase, and more, is expended in this layer of gas, heating it to a very high temperature, and thus bringing about the appearance of the spheroidal state. Some of the electrolyte may be volatilized, too. But as a rule in using this apparatus at as low a temperature as possible (in order to obtain a good yield) only part of the crucible is "open," *i. e.* in contact with *fused* electrolyte, the rest being frozen. Now when the anode effect takes place, the "open" surface of the crucible, and the electrolyte next it, are heated very rapidly, hence the adjoining electrolyte in contact with the crucible is thawed and the "open" or active surface of the anode (crucible) is increased. But at the higher temperature now prevailing at the anode, the critical current-density is much lower, hence quite a little of the congealed electrolyte must thaw out before the critical point is passed and the layer of gas breaks. This again allows quiet electrolysis to take place, and on a larger anode surface than at first, hence below the critical density. The melt now cools slowly, gradually decreasing the active anode surface until the effect again appears.

To show the variation in power consumed, it is only necessary to calculate the watts before, during and after any effect. Taking, for example, one of the effects shown in the figure, the power at 5:14 is 1.9 kw., at 5:15:25, when the effect commences, it jumps from 1.75 to 3.7 kw., at 5:15:50, when it stops, the power drops from 3.6 to 1.63 kw. That the resistance of the gaseous layer is gradually dropping off from the beginning to end of each effect, is seen by inspection of the chart of any of the effects, and is readily accounted for by the increase of active anode surface.

If the current density is the controlling factor, it ought to be possible, by increasing this at any point, to cause the effect to occur. This was easily accomplished: all that was necessary was to momentarily swing the cathode toward the active anode surface, thus altering the lines of current distribution, and the effect could generally be produced at once. All effects marked with an X were thus produced. This method was not always successful, especially if attempted too soon after the close of the previous effect, and some of the effects thus produced disappeared almost before they could be recorded; but in general, when the electrolysis was in this unstable condition the effect could be produced at will.

There remain two peculiarities of the effect to be discussed: (1) that it was never noticed with a new crucible, or, as noted by Willner, could be prevented by changing the anode; (2) that if with the same apparatus, the current used was raised to 175 180 amperes, no trace of an anode effect was found. The first is explained by the fact that the crucibles when made did not have a smooth interior surface, and the irregularities, extending into the electrolyte, prevented the formation of the film of gas. As the crucible was used, these were gradually worn off, and the surface became noticeably smoother, allowing the film to form. With the higher current, the temperature of the whole electrolyte was much higher; in fact, almost all the upper portion of the crucible was "open," and the critical current-density was never reached. As an example of the opposite sort, in one run in the Borchers furnace a carbon rod, two inches in diameter, was used as anode, the cathode being of molten lead in the bottom of the apparatus. The anode effect began as soon as the furnace was

started, and persisted through the whole run, a current of about 200 amperes being used. Here there was no chance for an increase of anode surface as the temperature rose, hence the effect was permanent. Willner's results, showing that, contrary to the statement of Woehler, chlorine is still evolved during the effect, were confirmed by this run. As the Borchers furnace was too large to operate in the empty wind-furnace usually used, a hood was improvised to take care of the chlorine. So much escaped, however, as to make the furnace-room almost unbearable, and convinced us that, whatever else it might be, the combination of the chlorine with the anode could hardly be the cause of the effect.

Touching the anode-surface with the point of a file did not stop the effect, as it should have, according to Willner. Perhaps the much larger amount of energy involved (Willner used a maximum of 25 amperes) made the cooling effect of the file negligible. However, in a crucible that had been worn thin by oxidation, it was found that scraping off some of the loose graphite on the outside of the crucible stopped the effect at once, probably on account of the increased radiation and the cooling effect of the scraper.

With the upper part of the crucible protected, as described above, not only from the air, but also from the cooling effect of radiation, the temperature within the crucible was much more uniform, and more of the anode surface was open or active; hence with the same current there was less likelihood of the effect taking place, unless the supply of electrolyte ran short, and the electrolysis took place too low in the crucible, where it was more subject to the cooling effect of the coil in the bottom.

A few other points of interest may perhaps be mentioned. We did not wish to put fluorspar in the bottom of the crucible as recommended by Goodwin and Tucker and Whitney, since the commercial article is not very pure. The only purpose in so doing, as far as we can see, is to keep a frozen or insoluble layer on the bottom of the crucible and so insure the liberation of the chlorine at the side instead of directly under the metal. Using pure calcium chloride, no difficulty was found in freezing a layer of one-half to one inch in thickness, so that we used no fluorspar.

Willner, using the electrolyte of Ruff and Plato, was much troubled by the formation of a thick froth over the electrolyte, which he ascribed to the fluorspar used; we had the same experience with the electrolyte used in the Borchers furnace, the froth causing great bubbles to solidify over the electrolyte, and then be pushed over the edge of the crucible by fresh bubbles below, thus wasting the electrolyte, interfering with the feeding of fresh chloride, and facilitating the recombination of the calcium with the chlorine. This furnace had been made with a fluorspar bottom. We did not experiment further with fluorspar in the electrolyte, though at the time we were inclined to blame the lime, formed by the burning of the calcium, for part of the trouble. Analysis of this pasty electrolyte showed an alkalinity, as CaO, of 4.22 per cent.; Fe_2O_3, 0.07 per cent.; and free carbon 1.28 per cent.

As it seemed possible that the presence of this amount of lime might raise the melting point of the electrolyte, a series of melting-point determinations of lime-calcium chloride mixtures was made. The chloride was melted down in a crucible of Acheson graphite in a resistance furnace, and portions of lime made from marble were added. The thermocouple was of the platinum-rhodium type, and the galvanometer had a resistance of over 200 ohms. They were calibrated together frequently, using as fixed points the b.pt. of sulphur, 444.7°, the freezing point of antimony (Kahlbaum's C. P.), 630°.5, and the freezing point of electrolytic copper, 1084°. (Bureau of Standards, Circular 7). The melting point of pure calcium chloride is given by Ruff and Plato[35] as 780°, while Arndt[36] found it to be 774°. No analyses of the salts used are given, but they were probably very pure. On taking the melting point of two samples of our chloride we found (*a*) 766° and (*b*) 755°. No analysis was made of the first sample, but the second showed 0.66 per cent. CaO and 0.15 per cent. $CaCO_3$. The sample was taken from the melt after the determination, and as further work showed that more oxide lowered the melting point, our figures are easily explained. By adding successive portions of oxide, we were able to reduce the freezing point to 705°, where the analysis showed CaO, 6.24 per cent., $CaCO_3$, 3.38 per

[35] Ber., 36, 2357; Chem. Centr., 1903, ii, 413.
[36] Z. Elektrochem., 12, 337; Chem. Centr., 1906, i, 1817.

cent. With more oxide, we began to get a partial freezing at about 770°, while the rest of the melt did not freeze till 705° to 720°, indicating the presence of eutectics. On account of the presence of the carbonate, in quite large quantities, thus unsettling the results, it is not deemed advisable to publish them in detail, but they will be checked up by others in this laboratory, and published later.

Most of the previous workers have melted the calcium chloride in fire-clay crucibles before adding it to the electrolyte, although it is thereby contaminated. It is impossible to add the cold chloride to the electrolyte, as the moisture which is absorbed causes violent spattering; but we have found that if heated for a few minutes in an iron crucible over a four-post burner, even calcium chloride that has served for a long time in a desiccator may be added with impunity. It is not necessary or desirable to melt the chloride; heating at a low red heat is sufficient.[37].

There is one other point worth mentioning in connection with the apparatus of Tucker and Whitney. For a cathode, they used gas-pipe, with one end capped with as thin a cap as possible. We have not found this satisfactory, as the cap is gradually corroded more or less, and if an arc be inadvertently drawn between it and the electrolyte, it is likely to burn through, letting the water into the molten chloride. For a cathode, we prefer one-inch pipe, fitted with a coupling into which a solid plug is screwed. If this starts to leak it does so more slowly, and the consequences are less serious. Alternating current may be used to melt down the charge on starting, without much corrosion of the pipe, and reduces the contamination of the bath by oxide from the burning of the metal formed when direct current is used.

University of Minnesota.
October 13, 1909.

[37] See also Weber, Ber., **15**, 2316; J. Chem. Soc., 1883, **44**, 151.

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PREPARATION AND PROPERTIES OF CALCIUM CARBIDE.

M. de Kay Thompson.

The first attempt to estimate the equilibrium pressure of carbon monoxide in the monovariant system consisting of this gas, lime, carbon and calcium carbide was made by Rothmund.[1] By heating a mixture of lime and carbon in a carbon tube, in the atmosphere, to different temperatures for a few minutes, cooling and testing the charge for carbide, the temperature at which carbide was formed under these conditions was found to be 1620° C. This temperature, of course, would be dependent on the pressure of carbon monoxide, which would be one-third of an atmosphere, for the oxygen of the air would be nearly completely changed to carbon monoxide when equilibrium is reached.[2] This pressure, however, might have been less than this amount, as equilibrium might not have been reached in these experiments. Similar experiments were repeated five years later by Rudolphi,[3] who found the temperature corresponding to atmospheric conditions to lie between 1800° C. and 1819° C., that is, about two hundred degrees higher than Rothmund's value.

The object of the following investigation was to measure the pressure of carbon monoxide in this system for a series of temperatures, from which both the free energy and the total energy or the heat from the reaction could be deduced, and incidentally to compare the results with the above values. These experiments have not been completed, but, on account of the recent appearance of an article dealing with the same subject by C. A. Hansen,[4] it is thought best to publish the results so far obtained.

Apparatus and Method of Procedure: The apparatus used was

[1] Z. f. anorg. Chem., **31**, 136-145 (1902).

[2] This is incorrectly given by Rothmund as 1/5 at., the same as of the oxygen itself. When the oxygen combines with carbon twice its volume of CO will be produced, which fact was overlooked by him.

[3] Z. f. anorg. Ch., *54*, 170 (1907).

[4] Electrochem. and Met. Ind., **7**, 427 (1909).

an Arsem furnace as described by Hansen.[5] It was connected to that end of a syphon barometer which is usually open to the atmosphere, and to a Geryk pump. It was planned to place an Acheson graphite crucible filled with a mixture of lime, graphite and calcium carbide in the helix, heat to a definite temperature measured by a Wanner pyrometer, and read off the pressure on the mercury column. In order to approach the equilibrium from both sides, bottles for generating carbon monoxide from potassium ferrocyanide and concentrated sulphuric acid were connected to the furnace through two soda-lime bottles and a phosphorous pentoxide tube.

In some experiments only lime and carbon or calcium carbide were used, trusting to the reaction to produce the phases that were lacking.

The carbon screen ordinarily used to prevent too much loss by radiation was left out in experiments carried out below 1600° C. on account of the property carbon has of absorbing gases, and consequent uncertainty in the meaning of the pressure readings. The furnace was always evacuated to a pressure of less than a millimeter before beginning the experiment.

Before taking up the individual experiments, the condition of the inside of the furnace after a run should be described. At temperatures from 1500° C. and upwards there was invariably a white powder covering the inside walls and pipes forming the electrodes. This is a very important observation, as it points to the occurrence of a reaction that was not anticipated and which, up to the present, has prevented the author from obtaining any satisfactory determinations. This is the direct decomposition of the carbide according to the equation

$$CaC_2 = Ca + 2C,$$

and further combination of the liberated calcium according to the reaction

$$Ca + CO = CaO + C.$$

It is evident that if this is the true explanation of the origin of the white powder, the pressure of the carbon monoxide will be reduced below what it should be for equilibrium, and the con-

[5] See also Arsem, Trans. Am. Electrochem. Soc., 9, 153.

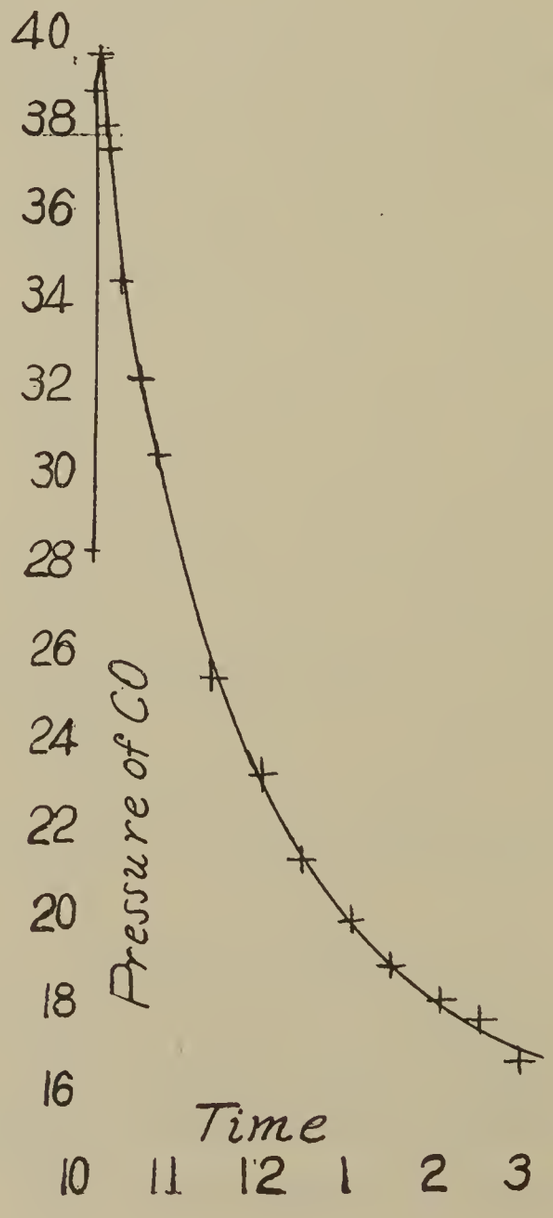
stant readings will be purely accidental. The evidence in favor of this explanation will be given in a description of some experiments. First, it will be shown that carbide breaks up in this way, in a vacuum. This fact has been assumed by Hansen and others,[6] but has not been proved as in the following experiment.

Experiment 5: Some carbide was made from Acheson graphite and Kahlbaum's lime in an arc furnace consisting of a graphite rod and crucible. This was crushed up and irregular shaped particles used varying approximately from ⅛″ to 1/16″ in largest dimensions; 25 grams of this were placed in the crucible and the furnace evacuated to 1 mm. pressure while cold. Radiation screen in position. When heating was begun the pressure started to rise at once, probably due to gases in the screen. When the temperature registered 1692° C. and the pressume less than 1 mm., on looking in the furnace the particles could be seen being thrown violently out of the crucible; even a small graphite plate placed on the carbide was thrown out. The heating at this temperature lasted less than an hour. When the furnace was cooled down and opened forty-eight hours later the contents of the crucible appeared to be graphite only and calcium was deposited over the inside of the furnace in a thin sheet. This caught fire very easily if brought in contact with a little moisture. This is not a property of massive calcium, but was probably due to the thinness of the sheet. This experiment shows then beyond a doubt that calcium carbide is decomposed in a vacuum at 1692° C. When, however, the experiment is carried out exactly similarly except that carbon monoxide is admitted, in place of the calcium metal the white powder is formed, or if the mixture contained some lime and carbon which would produce carbon monoxide, the same result was obtained. This was verified by a number of experiments. These experiments then prove the reaction between calcium and carbon monoxide. It probably takes place as the calcium comes off, in the form of vapor.

The fact that carbon monoxide unites with the calcium is shown also by the drop in pressure when the carbide and the gas are heated together and when at the same time the white powder is found on the interior walls after the run. In this case the appearance of the charge in the crucible is entirely different from

[6] See Abegg's Handbuch der anorganischen Chemie, Vol. 2, part 2, p. 120.

what it is in the case of the gas being absorbed with no white powder produced on the walls. When the powder is produced the contents of the crucible appear collapsed and look like graphite, while if the carbon monoxide is absorbed without the production of white powder it swells up, and in one case it stuck together and split open the crucible. This latter case is due, of course, to the reaction $CaC_2 + CO = CaO + 3C$. The densities of all three solids are not very different, and 64 gr. carbide changes into 80 gr. of lime and carbon, necessitating a volume increase. This is illustrated by the following experiment. The charge was twenty-five grams of carbide in pieces about half the size of grains of rice. Radiation screen not used. Furnace pumped out to 0.7 mm. and carbon monoxide let in to 28.5 cm. when cold. The following table and plot give the experiment in full.



Time	Current	Volts	Pressure in cm Hg at room temp.	Temp. Centigrade
10.13	0	0	28.5	20
.15	83.00		39.0	
.20	126.	45	39.9	1120
.22	129.	46	38.3	1169
.25			37.7	1169
.35			34.7	1169
.45			32.4	
.55			30.7	
11.35	131.	48	25.6	1169
12.05			23.4	1169
.35			21.5	
1.05			20.1	1169
1.35			19.1	
2.05			18.3	1162
2.35			17.9	1165
3.00			16.8	1180

The following experiment will show how completely carbon monoxide can be removed by metallic calcium. A mixture of 56 parts of powdered lime with 36 graphite was mixed with an equal quantity of powdered carbide. This was placed in the crucible in the furnace and the latter evacuated. Heating and pumping were carried out simultaneously till a pressure of 1 mm. and temperature of 1300° C. were reached with the pump shut off. The temperature was then raised to 1400 when the pressure fell to a perfect vacuum, as nearly as could be read on the scale. The scale could be read to only 0.1 mm. Simultaneously the brilliant light of calcium vapor could be seen in the furnace. It is here assumed that the gas giving the 1 mm. pressure to start with was carbon monoxide from the lime and carbon in the mixture. This experiment also shows that carbide is decomposed more rapidly than it is formed at this temperature and pressure. The following experiment when the charge consisted of only calcium carbide gave a similar result. The furnace was thoroughly pumped out, carbon monoxide admitted, and the temperature raised to 1735°. At first the gas expanded, and then the pressure fell from 34.8 cm. at 12.15 P. M. to 1.8 cm. at 1.55 P. M. On opening the furnace the usual white powder was found and the contents of the crucible appeared to be only graphite. These experiments show, then, the two different ways in which carbon

monoxide can be used up. It was further found on heating powdered lime and carbon at 1500° C. in a vacuum that the powder was found on the walls of the furnace, and no carbide in the crucible, thus confirming the above result obtained at 1400°, that under these conditions the carbide breaks up as rapidly as it is formed.

These results show that equilibrium measurements cannot be obtained quite as easily as was anticipated, from which it follows Hansen's measurements cannot correspond to equilibrium conditions. The only sure way to get equilibrium is to approach it from both sides, and this was not done in the single experiment quoted by him. Hansen's results may also be tested from a theoretical standpoint. According to the integrated van't Hoff equation the relation between the heat of this reaction Q, and the two pressures of carbon monoxide p_1 and p_2 corresponding to the absolute temperatures T_1 and T_2 is

$$4.57 \log_{10} \frac{p_2}{p_1} = Q \left(\frac{1}{T_1} - \frac{1}{T_2} \right).$$

Q at room temperature can be obtained as follows:

	CaO	$= Ca +$	O	$-$ 140,000 cal.[7]
$Ca +$	$2C$	$= CaC_2$		$-$ 7,250 cal.[7]
$C +$	O	$= CO$		$+$ 26,100.[8]
$CaO +$	$3C$	$= CaC_2 + CO$		$-$ 121,150 cal.

Approximately 121,000 cal. would then be absorbed by this reaction at room temperature. In order to know what the heat would be at 1600° C. the difference in the heat capacities of the substances produced and of those disappearing must be known.

The molecular heat of lime does not seem to have been measured, but can be calculated by taking the sum of the atomic heats of calcium and of oxygen.[9] The molecular heat of lime is then

[7] Landolt-Börnstein Tables, 3d ed., p. 436.

[8] *Ibid.*, p. 430. In the second and third reactions the carbon is in the form of diamond, but the molecular heat evolved in changing from graphite to diamond is only 500 cal., which small difference is of no consequence for the present purpose. See tables quoted, p. 464.

[9] Nernst, Theoretische Chemie, 4th ed., p. 180.

9.69, and three times that of graphite is 17.1, the total being 26.8.[10] That of calcium carbide would be 18.6 and of carbon monoxide at constant volume is 4.85, the total being 23.5. The difference in heat capacities then is 3.3 cal. per degree. This calculation is not exact, because all the specific heats used were not measured over the range of temperature here considered. These capacities, however, would probably not change very much, showing that the heat of the reaction would be changed at 1600° by approximately

$$3.3 \times 1600 = 5280 \text{ cal.}$$

giving about 116,000 cal. absorbed.[11]

It is possible with this value to calculate what the ratios of the pressures of carbon monoxide for any two temperatures should be. Substituting in the van't Hoff formula $Q = +\ 112{,}000$, $T_1 + 1{,}400 + 273$, $T_2 = 1{,}800 + 273$ the ratio of p_2/p_1 for these temperatures comes out 840, that is, for a rise of 400° the pressure increases 840 times. The ratio of the pressure obtained by Hansen for these temperatures is about 3.9. This, then, is another indication that the pressures observed by him were not determined by the equilibrium of this reaction.

It is intended to continue this work in the hope of getting the equilibrium values by modifying slightly the method.

Summary.

1. It was shown experimentally that the equilibrium pressure of carbon monoxide cannot be obtained by heating lime, carbon and carbide or lime and carbon alone initially in a vacuum, on account of the decomposition of the carbide into calcium and carbon and the subsequent reaction of carbon monoxide and calcium.

2. Hansen's results cannot, therefore, represent equilibrium. This was further demonstrated from theoretical considerations.

Electrochemical Laboratory,
Massachusetts Institute of Technology.
October 19, 1909.

[10] The values taken from Landolt-Börnstein Tables, 3d ed., are the following: Spc. ht. Ca = 0.1804, spc. ht. of oxygen at constant volume = 0.154, spc. ht. CO at constant volume = 0.173, spc. ht. graphite 0.475.

[11] The value given by Rothmund is 105350 cal. without giving the steps by which this value was obtained.

DISCUSSION.

Mr. C. A. Hansen (*Communicated*): I wish to state the circumstances leading up to my work on CaC_2, since I think they possess interest. When SiO_2 is heated in the presence of carbon, several distinct reaction products seem to be formed, which have been interpreted to indicate lower oxides of silicon. It seemed to me that since in the reaction $SiO_2 + C$ (if carried out in vacuo), CO would be the only gas phase, the determinations of the equilibrium temperature-pressure diagram should show the presence or absence of a series of distinct suboxides. To test out the method, however, I tried to determine the CO pressure of the simpler reaction $CaO + C_2$, since, so far as I know, calcium forms but the one oxide. The result of the unfinished work was published as a suggestion, since I found it necessary to drop this work for the time being.

I can confirm Mr. Thompson's note relative to the formation of a CaO film and of the pyrophorous Ca film upon the colder portion of the furnace, and I do not think it at all unlikely that reaction of calcium, with CO in the colder portions of the apparatus, prevents the determination of the true equilibrium pressure in this way at temperatures above which Ca is appreciably volatile, because it amounts to reaction taking place in the vessels at different temperatures for each of which there should be a distinct equilibrium pressure. While I knew that Ca was volatile at comparatively low temperatures, I was myself surprised to find its vapor pressure to be quite an appreciable quantity at 1500° C, and I discontinued the work at higher temperatures for this reason. It seems to me, however, that the failure of $CaO + 3C$ to react at 1450° C. when the CO pressure is 15 mm., and the fact that that reaction does take place at this temperature when the CO pressure is 3 mm. (as given), shows that at this temperature the equilibrium pressure is assuredly somewhere between 15 mm. and 3 mm. That reaction between CaO and C takes place at 1275° or under is also confirmed in my opinion by the following observation: When a compact pellet, made up with pitch, as described in my paper, is heated to 1200° C. or over in a very good vacuum, it crumbles, or at least becomes friable. If a similar pellet is heated in a CO atmosphere of a few mm.

pressure, it will not crumble. The pellet heated to 1450° at 15 mm. CO pressure was not in any way distinguishable from a similar pellet which had not been heated above 800° C. In other words, instead of being friable and crumbly, it was every bit as compact and strong as a piece of commercial electrode carbon, which it very much resembled. These phenomena must be due to oxidation by CaO of the binder carbon.

I do not consider the removal of the radiation screen a necessary precaution: First, because if one measures the CO pressure at equilibrium, the screen is saturated with CO and does not disturb the equilibrium; second, because removal of the screen makes a more non-uniformly heated heated reaction chamber; and, third, because at the crucible temperature of 1400° or over, the absorption of gas by the screen is not a very serious matter, since the coldest portions of the screen are well above a red heat. Also, as regards the use of an optical pyrometer in this furnace: The mica window of the furnace for which naturally the instrument must be calibrated by sighting at melting platinum or the like, becomes clouded more or less, so that the calibration of the instrument would need adjustment. I should suggest that the furnace be calibrated against an optical pyrometer if necessary as a check on the power-temperature diagram, but I prefer to use the diagram as more nearly reproducing temperatures satisfactorily.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 28, 1909; President L. H. Baekeland in the Chair.

THE PREPARATION OF SILUNDUM

By S. A. Tucker, H. F. Kudlich and E. M. Heumann.

Silundum is a name given to carbon articles which have been partially converted into carborundum in the electric furnace, and appears first to have been discovered by F. Bölling.[1] The method employed by Bölling for the production of this substance consists in heating the carbon in an atmosphere of silicon, the combination of the two producing carborundum, which is absorbed to varying depths by the carbon, depending upon the temperature and the length of time.

The conditions for the production of this material are only given in a very general way and it was therefore decided to make an experimental study of its formation.

Since it was evident that the reaction would depend upon the formation of silicon the first reaction used was that of F. J. Tone[2]:

$$SiO_2 + 2SiC = 3Si + 2CO.$$

A charge was made up according to this reaction and placed in a furnace of the general type of a carborundum furnace made of fire brick. The core instead of being of granular carbon, was made by pressing together a number of small carbon plates between two horizontal graphite electrodes the plates were 2½″ x 1¼″ x ¼″ pressed together face to face. Around their core was placed a charge of sand and carborundum in which was imbedded at varying distances from the core the carbon articles to be changed to silundum.

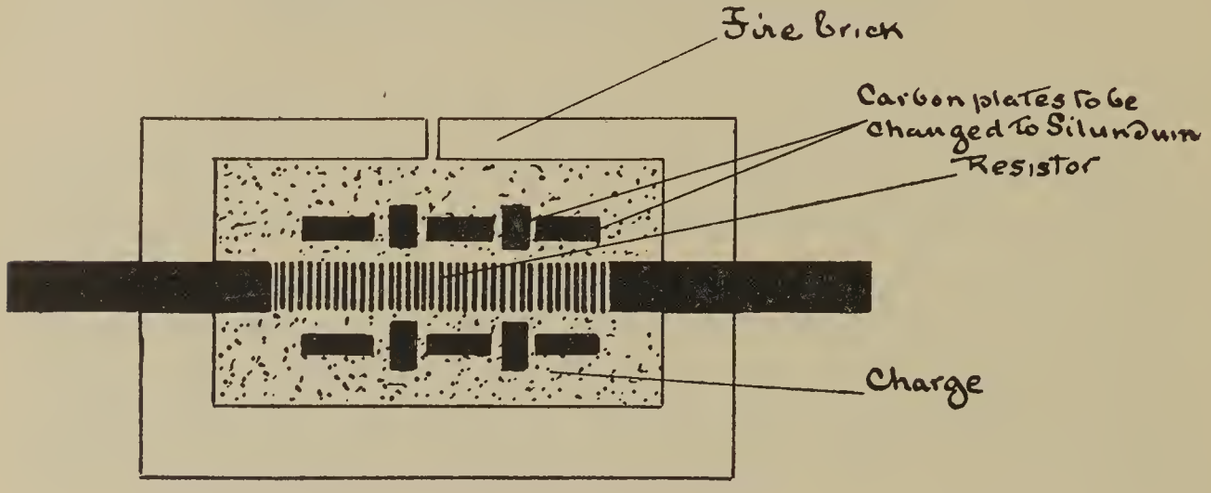
The side and end walls of the furnace were cemented as tight as possible in order to prevent the ingress of air which would tend to oxidize the silicon as it was formed. A current of 600 to

[1] F. Bölling, Chemiker Zeit., **32**, 1104.

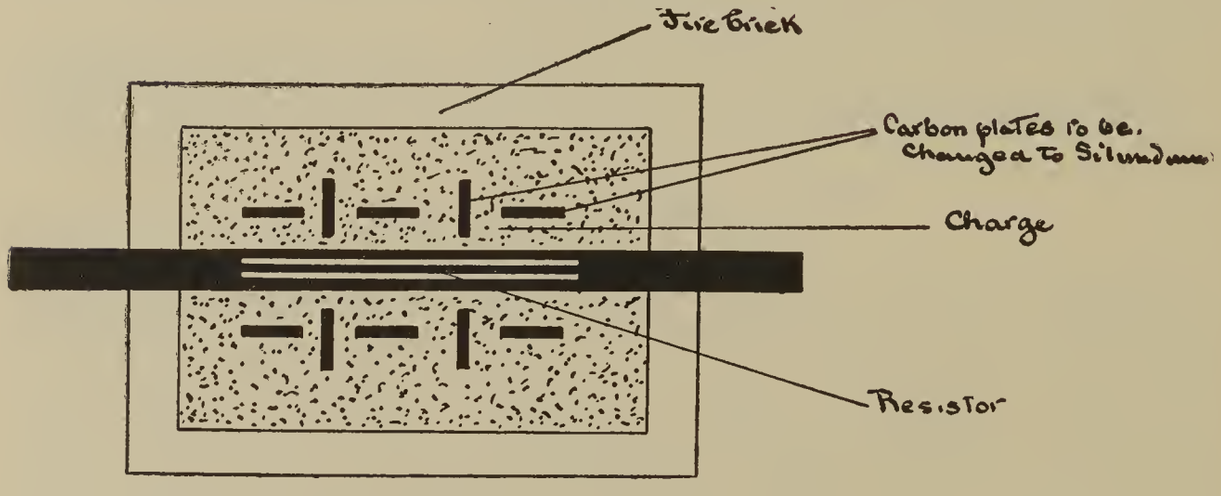
[2] F. J. Tone, "Production of Silicon in the Electric Furnace," Trans Am. Electrochem. Soc., **7**, 243.

700 amperes at 20 to 25 volts was kept up for 40 minutes, and was discontinued when the yellow flame of burning silicon was observed at the vent hole.

On cooling, some of the plates were more or less converted into silundum but the layer was only thin and it was decided that the temperature had been too high..



Silundum Furnace Elevation
Vertical Carbon plate Resistor



Silundum Furnace. Plan.
Horizontal Graphite Resistor

The next run was conducted in a similar furnace but less energy was used. At the beginning 2,400 watts were used, which was gradually increased to 6,000 watts toward the end as against about 15,000 watts for the first run. The duration of the experiment was about the same and it was found that the conversion of the carbon into silundum had penetrated to a greater depth than in the first case.

Run 3: Based upon the two previous experiments, a longer run was planned, the dimensions of the furnace being 9½ inches long, 8 inches wide and 7 inches deep. As before, the walls were carefully cemented and the top covered with fire brick. The core, however, was altered by placing three graphite plates lengthwise between the electrodes, the plates being held in place by the end pressure of the electrodes bearing upon them (Fig. 2).

About 5,000 watts were employed in this run for 3½ hours, the average voltmeter and ammeter readings being 8 volts, 600 amperes. Constant attention was required to keep the energy constant as the core became heated.

Very good results were obtained. Carbon plates placed directly underneath the core were almost completely silundumized while the plates above the core and at the sides showed a penetration of 1/16 inch to 3/32 inch, the plates being ¼ inch thick.

Run 4: Was a repetition of Run 3 excepting the graphite plates constituting the core were placed securely in groves made in the ends of the electrodes in order to prevent any tendency to arc at these points. The results were about the same. The carbon plates placed directly beneath and above the core were affected most, those on the side of the core receiving only a very thin layer (0.01 inch—0.02 inch) of silundum.

Another run was made following the conditions of the previous one, giving excellent results, plates and rods of carbon were all converted into silundum in varying depths depending upon their relative position to the core, those directly beneath the core showing the most complete conversion.

The energy conditions are given for this run in the following table:

Time		Volts	Amperes
Hours	Minutes		
	0	20	500
	10	15	540
	20	15	575
	50	10	620
1	05	10	635
1	30	10.4	625
2	00	10.4	620
3	00	10	620
3	20	20	610

The next experiments were made to investigate the reaction between sand and coke according to the following reaction:

$$SiO_2 + 2C = Si + 2CO.$$

Hence the charge was made by mixing 24 parts of coke to 60 parts of sand.

Run 5: This was made in the same manner as the last run, excepting that the above mixture was used as charge. It was only run for one hour as the resistor plates burned out. The voltmeter and ammeter readings remained fairly constant at 20 volts and 580 amperes. Even in this short time a thin layer of silundum was formed on many of the plates placed in various positions in the charge. The dimensions of the furnace were the same as that used in the former runs.

Run 6. In this run a granular carbon core was substituted for the plates. Owing to the high resistance of this core it was necessary to employ a much higher voltage. The run lasted one hour at which time the silicon began to burn.

Readings taken during the run were as follows:

Time Minutes	Volts	Amperes
0	180	0
10	160	10
15	60	125
20	30	150
55	45	200

The carbon articles placed in the charge were found to be unequally affected by the silicon. The faces exposed to the core were thickly incrusted and rough while the opposite faces were smooth. The core itself was found to be converted into silundum, although the penetration did not go far.

Run 7: The last run indicated too high a temperature, so in this the energy conditions were changed. The core was in this case composed of preheated granular petroleum coke of about 10 mesh (National Carbon Co.), which has a lower resistance than ordinary gas coke and allows of more easy regulation of the temperature.

The following readings were taken:

Time			
Hours	Minutes	Volts	Amperes
0	00	31	150
0	15	28	240
0	45	31	150
1	00	33	100
1	15	31	150
1	45	39	100
2	00	36	75
2	30	36	75

The presence of much undecomposed charge indicated either that the heat had been too low or that the time had been too short. Only the charge in the immediate neighborhood of the core had been affected. The carbon plates and rods directly underneath the core showed the greatest conversion, the other being scarcely affected.

Run 8: In this run, in which the most satisfactory results were obtained, no cover was built over the furnace, the gases being allowed to escape and burn over the entire surface as in a carborundum furnace. The dimensions of the furnace were 14 inches long, 10 inches wide, and 9 inches deep. The core being 11 inches long by 2 inches diameter.

The conditions maintained were:

Time			
Hours	Minutes	Volts	Amperes
	0	130	
	5	100	100
	10	42	250
	20	50	130
	40	46	225
1	20	40	250
2	00	40	250
2	45	40	250

The charge was more completely decomposed and gave a very good conversion into silundum of the plates and rods placed in it. The thin rods were in some cases completely converted into silundum, while all the rods and plates were transformed to the same material more or less completely. The proportions of the charge in this run were slightly changed, the proportions being 25 parts of coke to 60 parts of sand, in order to have a slight excess of carbon.

From these experiments it is evident that the material silundum can be produced by either of the two reactions used; the latter reaction, employing cheaper materials, would naturally be preferable. The temperature employed seems to be about the same as that for the preparation of carborundum.

The penetration or conversion of carbon into silundum depends upon the time of the run and also on the temperature employed. The conversion of the carbon into silundum depends upon its position with respect to the core. No very careful study was made of the product; the following points were noted, however.

In appearance it is compact, of a light grey to greenish color. It has a hardness apparently the same as carborundum and is heavy. This increase in weight over the carbon from which it is made is an indication of the more or less complete transformation of the carbon into silundum.

Thus in the last run the weight of two plates before and after conversion into silundum were as follows:

	Original Weight	Weight After Conversion	Difference
No. 1.	72.2 grams	90.7 grams	18.5 grams
No. 2.	74.4 "	95.0 "	20.6 "

Its most valuable property and probable use is its ability to stand a high temperature in the air without sensibly undergoing oxidation, and as it is a conductor of electricity, its use as a resistor material.

Several experiments were made with a view to testing it in this capacity, and they pointed to a satisfactory material for temperatures up to say 1500° C. In one experiment a plate of silundum was placed on a graphite block and pressed on its upper surface by a vertical graphite electrode. By passing sufficient current through this system the silundum plate was raised to a high temperature and maintained so for 3 hours. The graphite block and electrode were oxidized to such an extent that there was very little left, while the silundum plate was not affected at all excepting the surface exposing the carbon from which it was made, which happened to be broken off. The carbon here was, of course, burned away leaving a box-like shell of silundum. The temperature was read at various times and was found to be 1200° C. and over.

The electrical resistance of the material varies greatly, depending upon the penetration, but there seems to be no reason why the material should not prove valuable as a resistor material and come into general use for a variety of purposes in which a high temperature is to be maintained electrically.

Electrochemical Laboratory,
Columbia, University,
May, 1909.

DISCUSSION.

PROF. J. W. RICHARDS: This reaction recalls to my mind what I have tried to conceive as the principle involved in the preparation of carborundum. I have studied the carborundum furnace a good deal, and seen it in operation, and I have always thought that the large crystals produced were produced by contact of silicon vapor and carbon vapor. I could not conceive of the production of these crystals in place, except from the interaction of two vapors, one of silicon and one of carbon. Here is a case where we have the proof, in the electric furnace, of the presence of a large amount of silicon vapor, and I think that, at the proper temperature, in contact with carbon vapor, this causes the formation of the crystals of carborundum.

DR. WHITNEY: I want to find fault with the definition given by Dr Tucker. I do not know much about the silundum, but feel more free to talk about carborundum. The definition in the paper is: "Silundum is a *name* given to carbon articles which have been partially converted into carborundum in the electric furnace, and appears first to have been discovered by F. Bölling." Is that what silundum is? I would just like to know. I imagine silundum is a compound or mixture of some materials. Is it a mixture containing carborundum and carbon, or silicon?

DR. TUCKER: As far as I can see, there is a penetration of the silicon vapors into the massive carbon. This penetration depends, as I have said, upon the temperature and the length of time it is exposed to the effect. The converted material on

the outside is what Bölling calls silundum. As to what the chemical composition of that is, I do not know, because I have not analyzed it, and I do not think anyone else has. It has a good many properties of a very similar nature to carborundum. That is about all I can say.

Mr. C. A. Hansen: I would like to confirm Dr. Tucker's statement as to the resistance offered to oxidation by "silundum." We had a grid, such as Bölling showed a picture of in the *Electrochemical and Metallurgical Industry* last Spring, made up of 36 pieces of "silundum," each ⅛ inch diameter and 1 inch long, cemented together in series, apparently by fusing silicon around the junction. This grid was heated to 1300° to 1400° C. for two days, exposed to the air, and finally failed mechanically, not by oxidation. These rods, not including any of the silicon joints, analyzed 80.2 per cent. silicon, which is some 10 per cent. in excess of the silicon present in carborundum.

Mr. W. M. Kohn: I would ask if Dr. Tucker can give any data showing the relative resistivities of the silundum and the original carbon from which it was made. Is the resistivity greater or less?

Dr. Tucker: The resistance is greater, but every piece of the material we obtained seemed to have its own resistance, depending on the penetration of the silicon vapor, and as far as I could find out, those which were completely converted through and through were practically non-conducting at ordinary temperatures.

Mr. Hansen: Regarding the resistance of silundum, the grid I described had a resistance of 39.4 ohms at room temperature, 27.3 ohms at 700° C., 24.7 ohms at 1125° C. and 22.4 ohms at 1400° C. At 1400° C. it dissipated 5.4 amperes at 121 volts.

Mr. F. J. Tone: I have analyzed many samples of silundum made in accordance with the process Prof. Tucker describes, and in every case I found that it consisted principally of carborundum. The percentage of carborundum varied from 96 per cent. to 99.5 per cent. There is often present a small percentage of elemental silicon, but in all substantial respects the material is simply carbide of silicon in a dense, compacted form.

I do not agree with Prof. Tucker that pure silundum is a non-conductor. It has a high resistance when cold, but it has a large negative temperature coefficient, and its conductivity increases very rapidly with rise in temperature. I have found, as Prof. Tucker states, that penetration depends on the time of the run and the temperature employed. I think that another consideration which governs, more than either of these, is the character of the atmosphere in the furnace. A mixture of vapors of silicon and silica has a greater power of penetration than pure silicon.

Mr. F. A. Lidbury: It seems to me that the discussion which has already taken place furnishes one more proof of the inadvisability of the practice (which is unfortunately becoming more frequent) of making investigations of reactions involving changes of composition, without carrying out any analyses whatever on the resultant product.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 28, 1909; President L. H. Baekeland in the Chair.

THE TITANIUM ARC.

By Wm. S. Weedon.

Many years ago a series of experiments was started in order to determine which substance, among the materials available, would give the maximum light efficiency when used as arc electrodes. The work covered a large number of elements, compounds and mixtures, with considerable variation in the method of preliminary treatment.

Among the substances that were tried there was none which gave as high efficiency in candle power per watt, as material which contained the element titanium. Of these materials, the carbide of titanium can be obtained in the market in several forms, and as this is probably the most convenient and important material dealt with in this line of work, these should be given.

1. Carbide containing more carbon than is necessary to form the combination TiC, in which the excess of carbon is in the form of graphite.

2. Carbide containing only as much carbon as is necessary to form the carbide TiC, or less than this amount, the latter kind being in all probability a mixture of the carbide TiC and metallic titanium a small amount of iron also being present.

3. Carbide containing a rather low percentage of carbon and a fairly high percentage of iron.

In the early stages of the experiments the second class of material seemed to be the most satisfactory, while the third class gives an arc which possesses certain objectionable characteristics. In particular the presence of iron seems to cause unsteadiness. As is well known, metallic arcs are far from steady under ordinary circumstances, and in this case the iron seems to accentuate the tendency to unsteadiness.

Characteristics of the Titanium Arc: Direct Current.

The arc, as obtained from the carbide, possesses an extremely luminous inner path, while little light is given by the outer mantle and none whatever by the craters. To give the best results the titanium carbide electrode should be the cathode and placed below an anode of copper, the latter being inactive and not wasting appreciably. The light is almost pure white, only

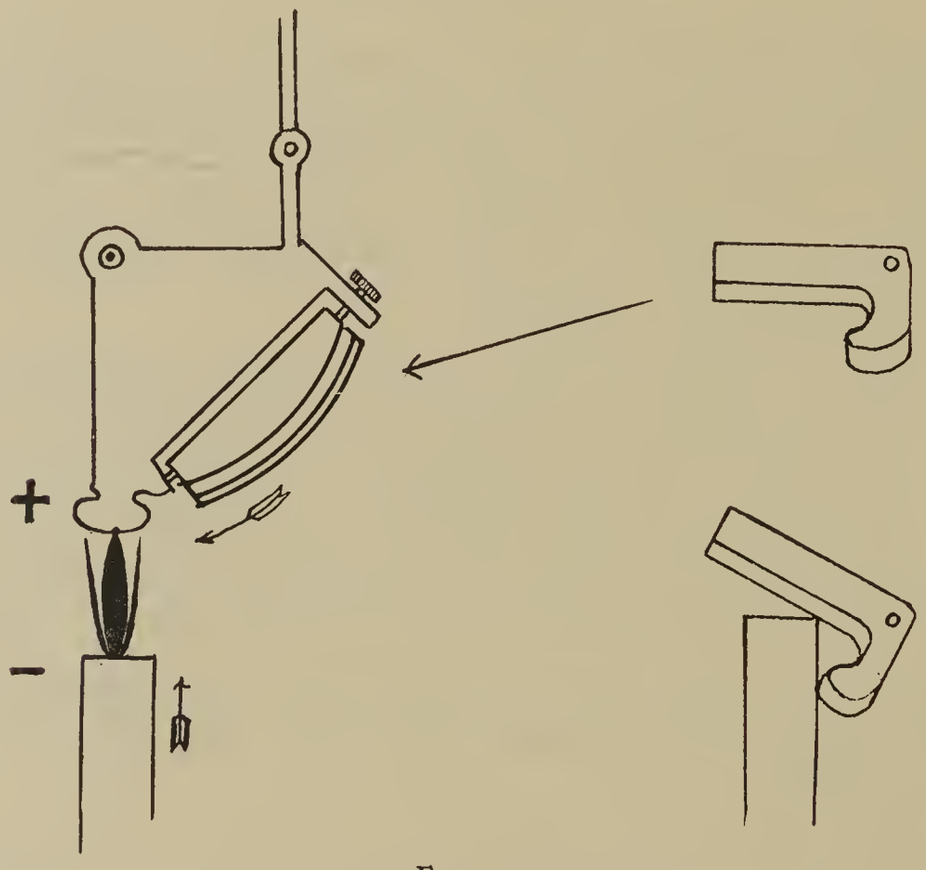


Fig. 1.

the faintest tinge of yellow being noticed when observed from a distance.

After running for some time, there is deposited a "button" of titanium oxide on the copper anode, which causes unsteadiness at times. The phenomenon known as "hissing" or "positive blow" occurs occasionally, resulting in the production of a peculiar jet of flame from the anode, in a consequent variation in light intensity, and in a decrease in voltage across the arc. This was discussed in a previous paper, from a theoretical standpoint.[1] Further, another difficulty is caused by the formation of

[1] Trans. Am. Electrochemical Soc. (1904), Vol. 5, p. 171.

an oxide layer on the surface of the titanium carbide electrode. It will be seen that this leads to complications in starting the arc after it has been extinguished for a short time, since the oxide is non-conducting.

The button on the anode does not offer serious difficulty, since a device by Mr. Halverson, designed for the magnetite lamp, overcomes this obstacle. The device referred to is an anode "rocker" connected with the lamp mechanism, so that the arc

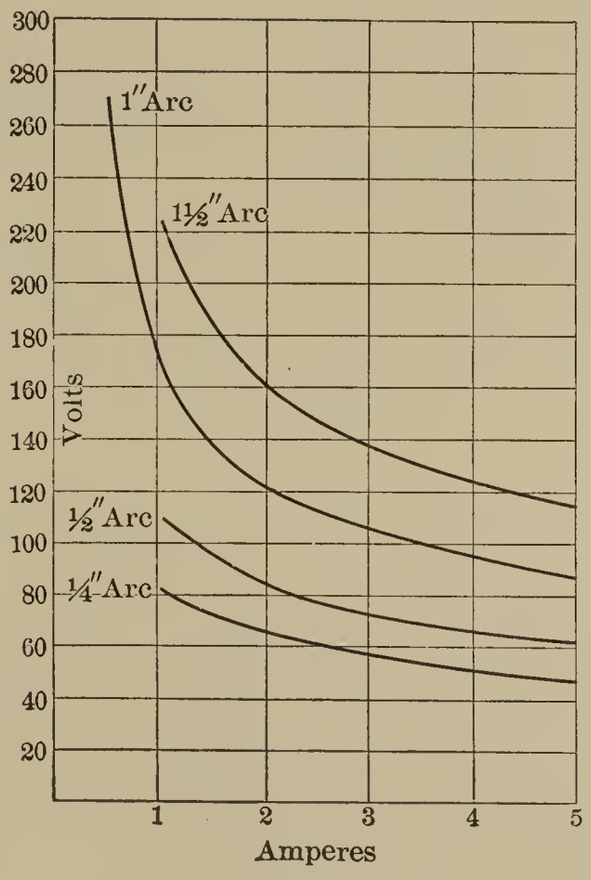


FIG. 2.

plays upon one portion of the anode while running, but strikes from another portion. By adapting this idea of the rocker anode, it has been found possible to strike the arc, even though the cathode and anode both have non-conducting surfaces. The starter and method of operation are shown in Fig. 1.

It will be seen that the arc is struck from the side of the lower electrode just below the tip, and after the current is established the electrodes return to running position, the arc running along the anode and climbing the cathode until it also is in normal position.

The titanium arc is a flame arc, and the efficiency increases

with increase in current density and with increase in arc length. The volt-ampere curves are given in Fig. 2, and the volt-arc length curves in Fig. 3. The power consumption curves, shown in Figs. 4 and 5, indicate that we have here a very efficient

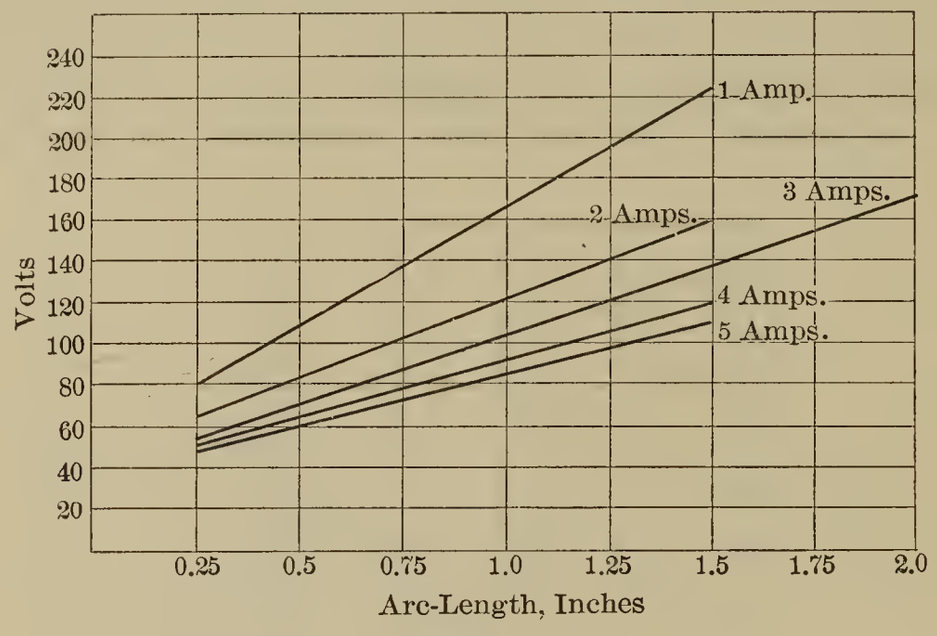


Fig. 3.

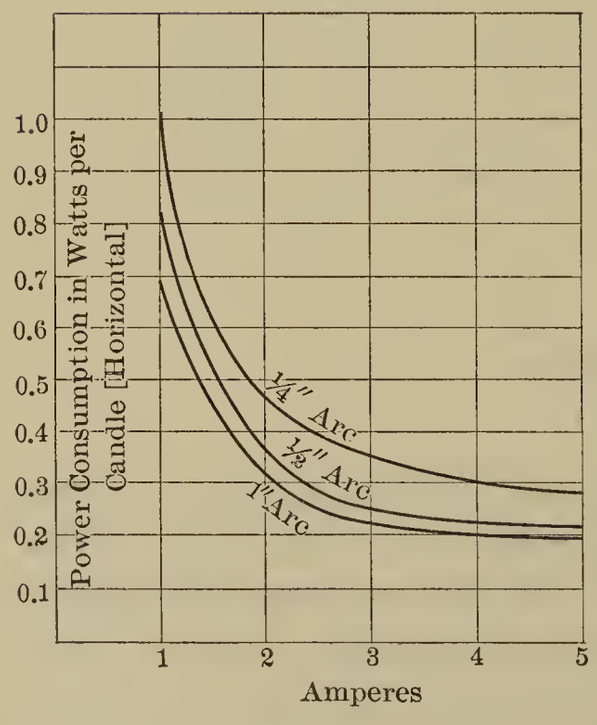


Fig. 4.

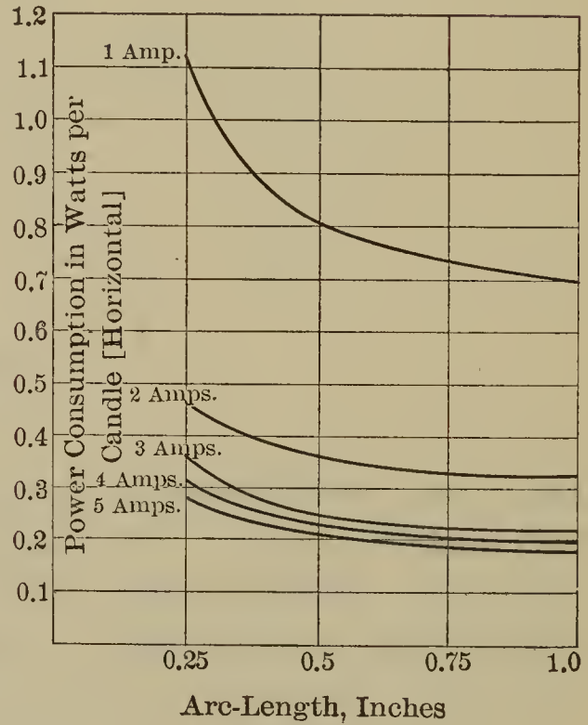


Fig. 5.

arc. At 3 amperes, 103 volts and 1 inch arc length, we obtain 1,355 horizontal candle power, giving a consumption of only 0.228 watts per horizontal candle power. The mean spherical candle power at 2.87 amperes, 95 volts (273 watts), is 535, the

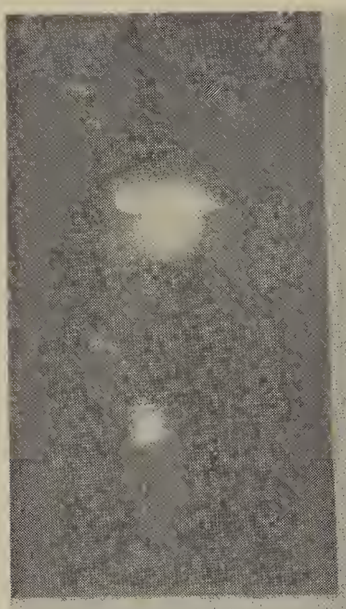


Fig. 6.

Fig. 7.

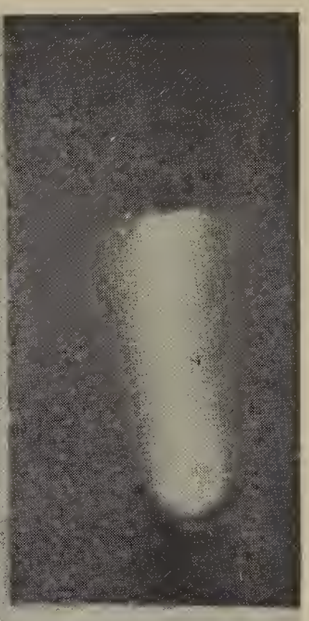


Fig. 8.

Fig. 9.

Fig. 10.

Fig. 11.

Fig. 12.

Fig. 13.

Fig. 14.

PHOTOGRAPHS OF THE TITANIUM ARC.

mean hemispherical candle power is 737.6, and the horizontal candle power in this case is 1,240. On this basis the power consumption is 0.51 watts per mean spherical candle power, and 0.37 watts per mean hemispherical candle power; this corresponds to an efficiency of 1.96 mean spherical candle power, or 2.70 mean hemispherical candle power per watt.

The electrical measurements given were made at the arc terminals, and the photometric measurements were made through a clear glass globe of the usual thickness.

A study of the efficiency curves will show that titanium carbide arcs are most satisfactorily operated on a constant current circuit rather than on a low voltage multiple circuit (with the necessarily short arc length).

Some photographs have been taken in order to give a comparison of the various arcs. Since these were taken through the globe, the reflection of the flame is sometimes reproduced in the photographs, and should not be confused with the arc itself.

Fig. 6 shows the plain carbon arc. Note the absence of light in the arc stream, while the terminals are very luminous.

Fig. 7 shows the arc obtained from Conradty's flame carbons. The arc stream is luminous as well as the terminals, but there is an entire lack of structure in the flame.

Fig. 8. Magnetite as cathode and copper as anode. Arc length 1 inch, current 3 amperes. No light is given off by the terminals, and all comes from the path of the arc. Structure shown clearly, the inner path giving a large portion of the light, but the outer mantle also luminous.

Fig. 9. The titanium arc. Lower electrode (cathode) titanium carbide. Upper electrode copper. Arc length 1 inch, current 3 amperes. Structure very clearly defined, inner path giving nearly all the light.

Fig. 10. Same as Fig. 9, but showing tendency for the outer mantle to form in layers. This occurs occasionally, and does not interfere with the steadiness of the arc.

Fig. 11. Titanium arc, flickering due to air currents. This arc is very sensitive, and the air currents must be controlled carefully by suitable draft arrangements.

Fig. 12. This shows the tendency of the titanium arc to "crawl" up the side of the anode, owing to air currents or improper centering of electrodes.

Fig. 13. The titanium arc is also sensitive to irregularities in the composition of the electrode. In this photograph a dark space is observed around the anode, and this causes flickering. The titanium used contained iron.

Fig. 14. This photograph is not very clear, owing to the confusion caused by the reflection in the globe. It represents the tendency to darken around the cathode, and this seems to be due to the presence of some impurity in the carbide. At times the dark space will appear, extending part of the way up into the arc from the cathode, and often will dance up and down

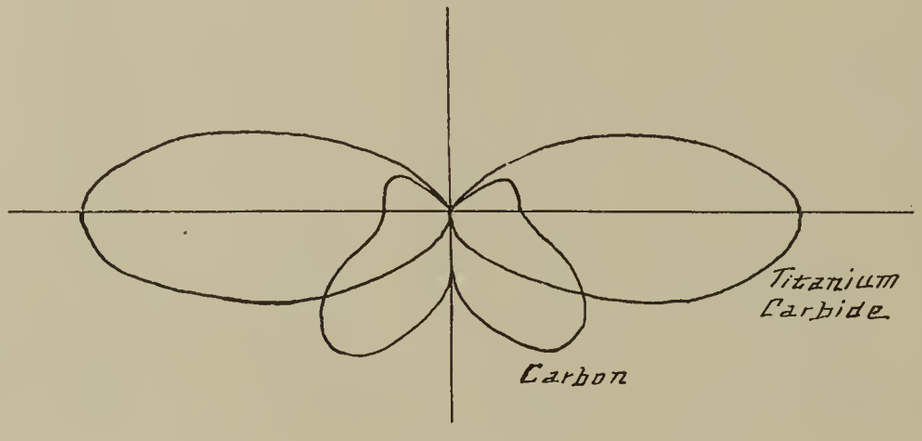


Fig. 15.

for some time. It differs from the darkening around the anode in that its limit is sharply defined. When pure carbide is used this does not often occur.

As is to be expected, the distribution of light from the titanium arc is quite different from that of the carbon arc. Fig. 15 will serve to show the difference in this respect, although the figure is not drawn to scale. It is to be noted that flame arcs are better for street illumination than the ordinary carbon arc, even at the same mean spherical candle power.

Preparation of the Titanium Carbide Electrodes.

For the initial experiments the electrodes were made as follows: The lump material is coarsely powdered and then pulverized

in a ball mill to a high degree of fineness. It was then worked in a roller mill with oil, glycerine or other suitable binder. After the material was thoroughly plastic it was extruded through a nozzle by means of a hydraulic press, the sticks being cut to suitable lengths.

After drying at ordinary temperatures, and then in a gas oven, the electrodes were fired at high temperature, preferably in an electric furnace of the carbon tube type. After firing, the sticks should be firm and without cracks. If fired at too high a temperature they are liable to be brittle.

It was at that time thought to be necessary to copper plate the electrode to prevent oxidation during burning in the lamp. This phenomenon is characteristic of titanium carbide. A bare electrode oxidizes at a point ⅛ to ¼ inch below the tip, the stick swells, and the top falls off. The copper plating effectually prevented this, and did not interfere materially with the running of the arc.

The life of the carbide stick varies, of course, with the current density and inversely with the diameter of the electrode. At about 3 amperes a stick ½ inch in diameter wastes away at the rate of 1/15 to 1/10 inch per hour.

During the running of the arc the whole of the upper surface of the electrode does not melt, but simply appears moist at the point at which the arc is playing. The more nearly the composition of the stick approaches metallic titanium, the higher the melting point, and it is certain, from a number of experiments made in connection with the preparation of these sticks, that metallic titanium is one of the most refractory metals known.

The titanium carbide arc may also be operated with the alternating current under certain conditions, but the results of this work cannot be presented at the present time.

The Titanium Sub-oxide Arc: Direct Current.

The material used in these experiments was prepared in the electric furnace by the reduction of rutile. Its preparation is quite easy, since the melting point of the product is much lower than that of the carbide.

The character of the arc is almost identical with that of the

carbide arc, but the light is much steadier and the tip of the electrode does not oxidize and fall off. It is therefore not necessary to plate the sticks or cover them with a metallic sheath.

There are certain objections to this electrode, and among these are the tendency to form large, troublesome deposits on the anode, and the fairly rapid consumption of the sub-oxide stick.

The sub-oxide gives a light efficiency even better than that of the carbide. At 3.4 amperes, 7/8 inch arc length and 100 volts, 2,100 candles (horizontal) are obtained. This gives a power consumption of 0.162 watts per horizontal candle power, or an efficiency of 6.17 horizontal candle power per watt. The distribution of the light is the same as with the carbide.

The heavy deposit on the anode is probably responsible for the fact that the difference in potential between the terminals is about 15 volts higher than is the case with the carbide at equal arc length and current.

The sub-oxide electrodes cannot well be operated with the alternating current, this being due to difficulties caused by rectification.

The above work was carried out in the research laboratory of the General Electric Company.

DISCUSSION.

Dr. W. R. Whitney: It is not that I rise to discuss the paper so much as perhaps to offer an apology. Most of this work which has been presented to you by Dr. Weedon was the result of original experimentation in the use of titanium in lamps, and I rise not to discuss the subject-matter presented in the paper, but to apologize for the delay in the publication of the experiments. It is difficult for a company like the General Electric Company, as I believe you will understand, to permit the early publication of the results of work of this class, obtained by men operating in the laboratory, and I think I am right in saying that Dr. Weedon had this paper written as early as 1904. He kindly held the paper in abeyance until its publication could be permitted. Our research laboratory would like to be able to publish

rapidly and immediately the results of its experiments, but the commercial conditions do not always permit this. If the laboratory published articles such as this one, for example, at the time the experiments were being carried out, the commercial conditions might be something as follows: The people would say: "Well, that new lamp is coming out now, and we want that lamp and will not buy any other." But that lamp may not work out at once to meet the conditions of public use; it may even fail totally, and, therefore, the results of the experiments cannot be published as promptly as might be desired. The work on this lamp has all been done as rapidly as it could be done, and yet there are only three or four cities which have in use the titanium carbide lamp, and the lamps are hardly in use in more than experimental numbers, 100 lamps or so in each case.

One interesting thing always appeared to me in Dr. Weedon's data, which I believe thoroughly, and that is the efficiency obtained in the arcs. I do not know whether you are all familiar with the question of theoretical light efficiency. One way of determining luminous efficiency is to submerge a lamp in a vessel of water and determine the rate at which it heats the water. The comparison of rates at which the lamp heats the water, when transparent in one case and opaque in another, gives the data for determining the light efficiency. That was determined by Tumlirz, and his 100 per cent. efficiency was found to be 0.22 of a watt per mean spherical candle power. This, Dr. Weedon has nearly reached in the titanium suboxide arc (0.377 of a watt). In other words, apparently, from this data, we are getting down nearly to the 100 per cent. efficiency for light. The probability is, there is an error in the Tumlirz measurements. Similar data obtained by Aengstrom gave 0.11 of a watt per mean spherical candle power as 100 per cent. light efficiency. This may also be still too high. The interesting question always comes up as to how nearly we are to the theoretically perfect light. You see, in that connection, that we still have about as far to go as we have gone.

Dr. S. A. Tucker: I have worked on this subject for several years, more or less, and I have tried a good many different compounds to make carbide, and have made them up into electrodes and tested them with a photometer. It may be that I did

not cover the ground in quite the same way that Dr. Weedon did, but my experience was that the zirconium carbide was more efficient than the titanium carbide. I would like some information on that.

Dr. Weedon: We tried the zirconium carbide obtainable at that time, and it was not as efficient as the titanium carbide.

Prof. J. W. Richards: A thought came to me as to whether the carbides of some other of the very rare metals might not be equally efficient, and I wish to ask Dr. Weedon whether they had tried the carbides of aluminium, magnesium, beryllium, cerium, lanthanum, didymium, zirconium, and thorium, or mixtures of the same, as to whether they might not give great luminosity in this arc.

I have read recently a paper in which it was stated that the heat of oxidation of these titanium compounds probably plays some part in the increased luminosity of the titanium arc, and I have made a calculation based on the rate at which this pencil wastes away. The heat generated by the oxidation of the titanium carbide amounts to only 1.7 per cent. of the heat generated by the electrical energy used, so that it can only cut a small figure in the total luminous effect.

I wish to remark also, in connection with this general subject, that my friend, Mr. Porter W. Shimer, of Easton, was the first one to describe titanium carbide, in a paper published in 1886, before the American Institute of Mining Engineers. He found, on dissolving pig iron, a residue of titanium carbide in perfect cubical crystals. He showed me specimens, gathered at a great deal of trouble, which he separated out, analyzed and found to be titanium carbide, having the proper formula. He came to the conclusion that titanium, as dissolved in pig iron, was there as titanium carbide, and not as simply diffused or generally dissolved titanium. That may have some bearing on the properties of titanium steels.

Dr. Weedon: We have tried, I think, practically all of these rare metal carbides, and we were not able to obtain any which had greater efficiency than the titanium carbide, but I think it should also be stated that a great deal depends on the direction of current used. For instance, thorium carbide, which gives

very little light, when used as a cathode, gives a characteristic luminous thorium arc when used as an anode. The probability is that the direction of current used may have something to do with the efficiency of these arcs as tried by Prof. Tucker.

DR. WHITNEY: One important suggestion to make at this time is that the use of the rarer metals is out of the question in arc lamps until we have some way of preventing the consumption of the electrode. The electrode will burn from one hundred hours to two hundred hours, and most of the metals which have been mentioned are too expensive to be practically used in electrodes which will burn for comparatively so limited a time.

DR. WEEDON (*Communicated*): In arcs of this kind I have frequently found it convenient to use a constant for converting figures for horizontal candle power into those for mean spherical candle power, and *vice versa*. The constant which I have used for the former is 0.43, and for the latter 2.32, which will be found to be sufficiently accurate for ordinary purposes.

Referring to the characteristics of the titanium arc, the statement made with regard to the fact that the inner path furnishes almost the entire light given by the arc is not strictly accurate, since the innermost path of the arc is practically non-luminous, and the very luminous path, which is quite narrow, possesses somewhat the form of a luminous tube surrounding this non-luminous space. The portion of the arc, however, which is generally referred to as the "outer mantle" furnishes only a very small amount of light.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909; President L. H. Baekeland in the Chair.

CONTRIBUTION TO THE ELECTRIC SMELTING OF IRON ORE.

By Edward R. Taylor.

The necessity for large furnaces for the electric smelting of iron ores will be upon us in the very near future. It is often the case that plans that serve for small demonstrations are not well adapted for large operations. In this connection the carbon bi-sulphide electric furnace now in use offers some suggestions that may be of use.

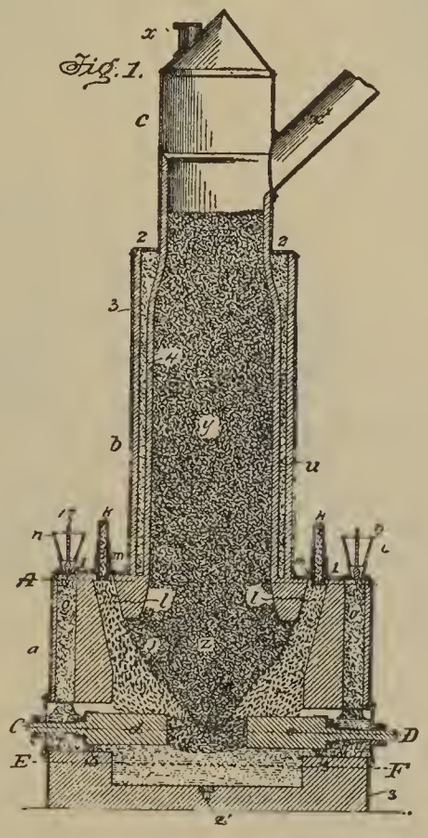
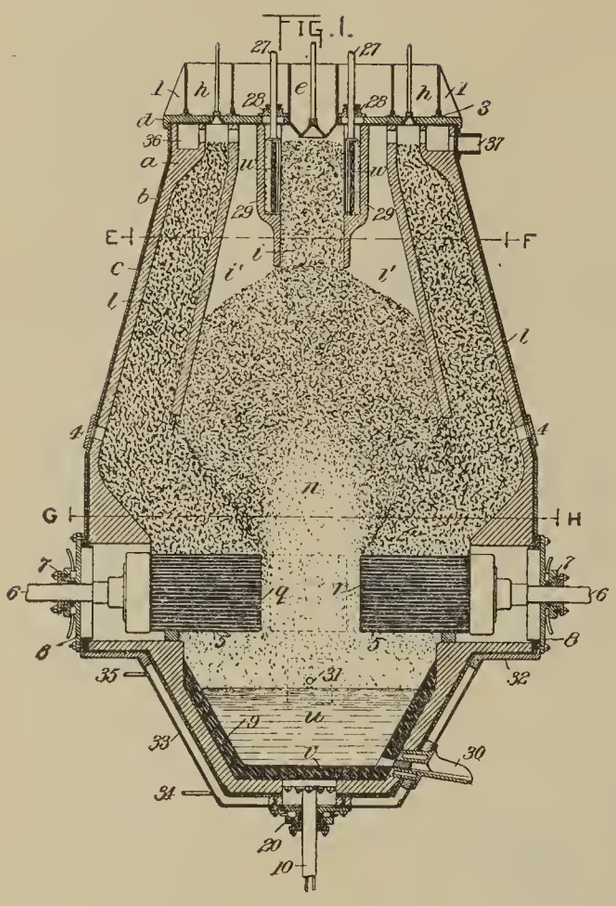


Fig. 1, with enlarged bottom, shows this furnace with electrodes and broken carbons for their reinforcement, to which your attention is invited. Several years' experience in the use of this means of prolonging the life and regulation of electrodes has demonstrated its great usefulness, and points to its value in furnaces of large size.

FIG. 1.



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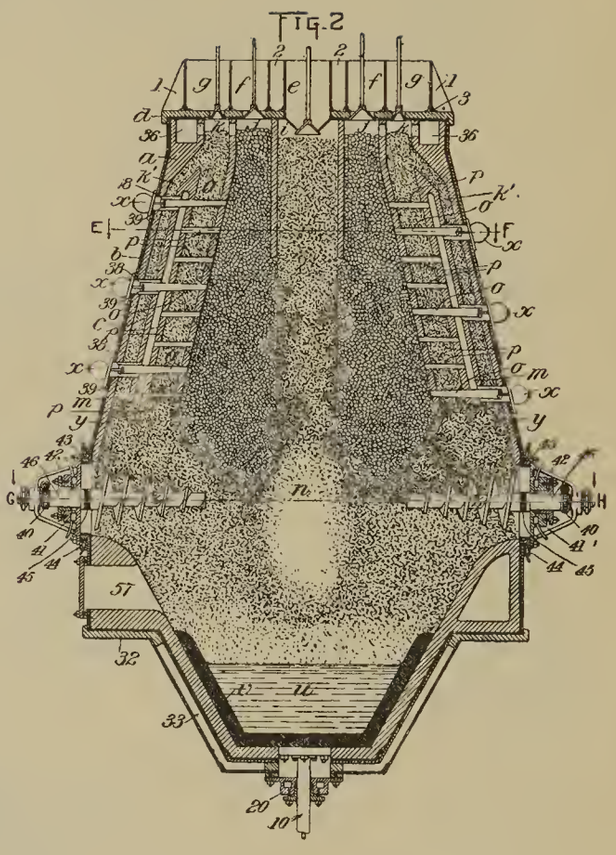


FIG. 3.

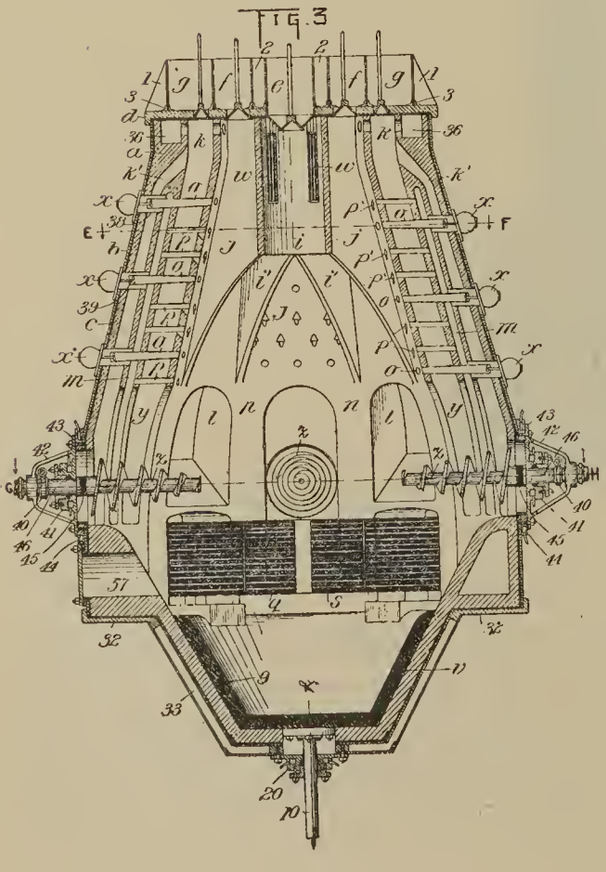
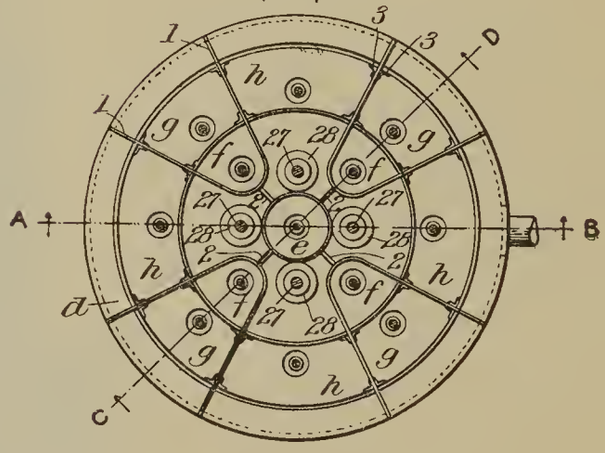


FIG. 4.



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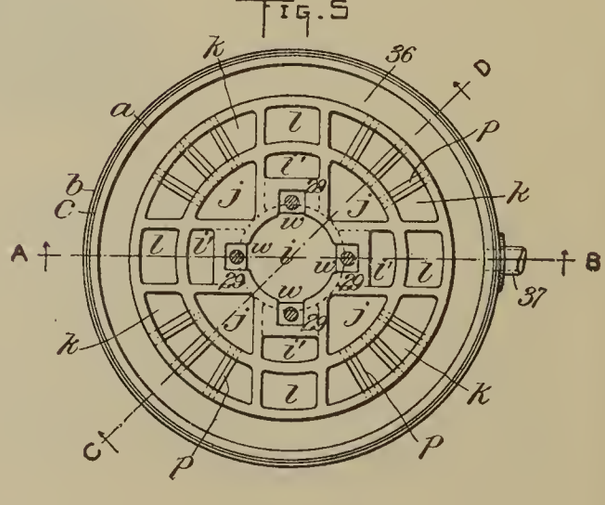
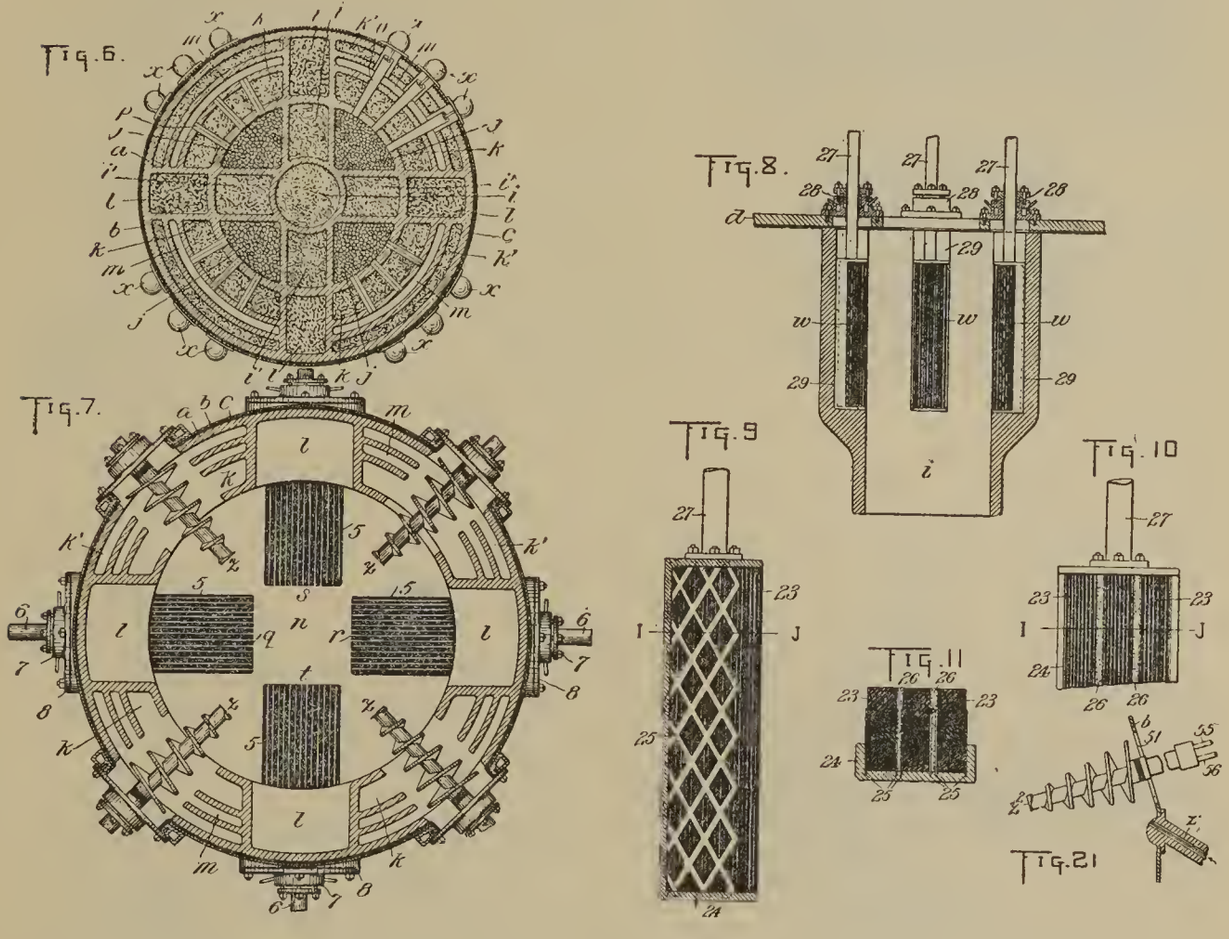


In the plans about to be described, it is hoped that a contribution has been made to the large-scale furnace for the electric smelting of iron ore. It also embraces an endeavor to make the incoming charge serve to conserve the heat by feeding the fine ore around the periphery of the furnace and subsequently forcing it towards and into the heat zone of the furnace.

The electrodes are placed under arches which protect them from the descending material and permit their renewal when necessary. These arches, and others protecting the feed screws



for forcing the fine ore into the interior of the furnace, serve to support the upper brick lining of the furnace. This is shown in Fig. 3. It shows a cylindrical construction resting on arches, through which charcoal or other form of carbon is fed to maintain a vertical column down to and between the main electrodes, which is designed in part to prevent the ore from occupying that space and so enabling it to form and be maintained as the weakest part of the electric circuit. That is that portion which is between the main electrodes. Outside of the cylinder just described is a

FIG. 12.

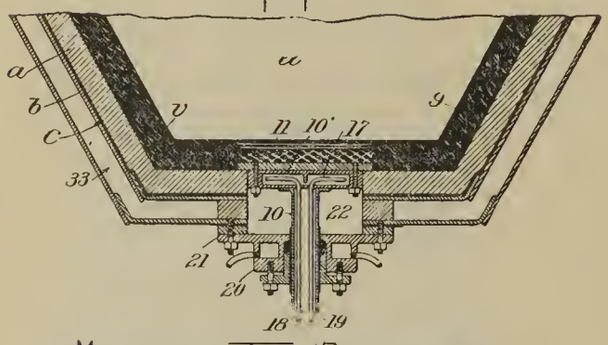


FIG. 13

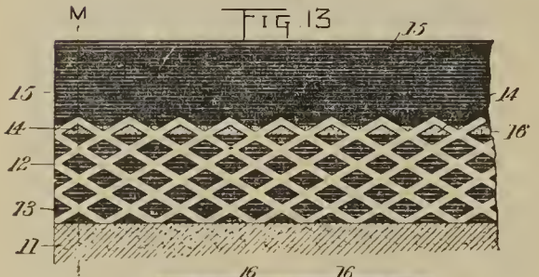
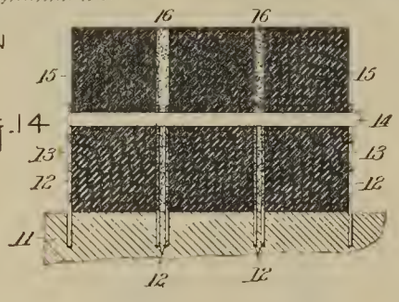


FIG. 14



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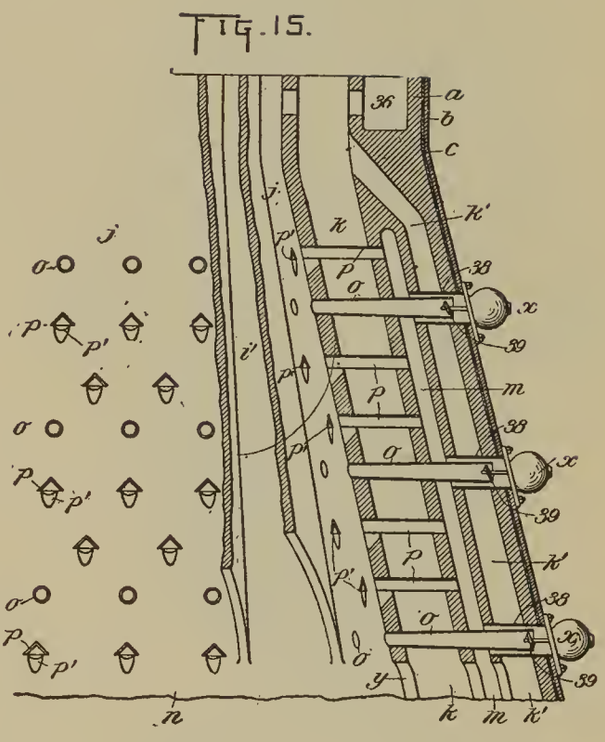

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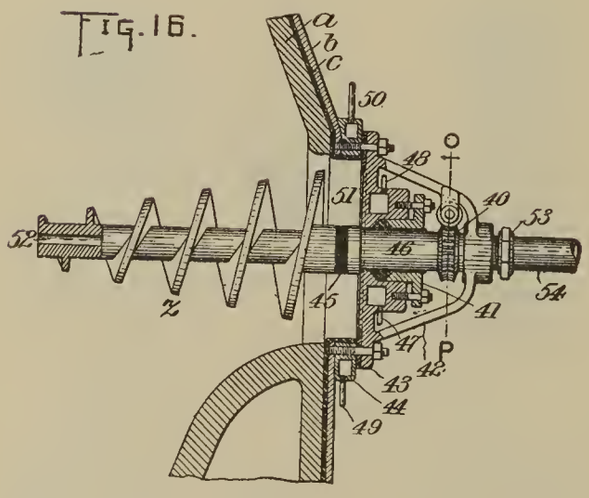
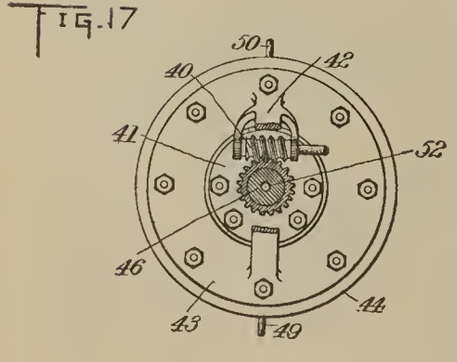


FIG. 17



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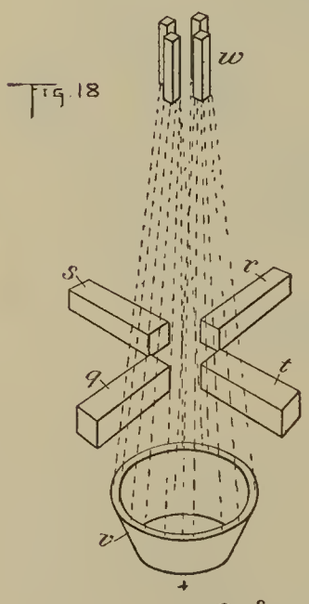

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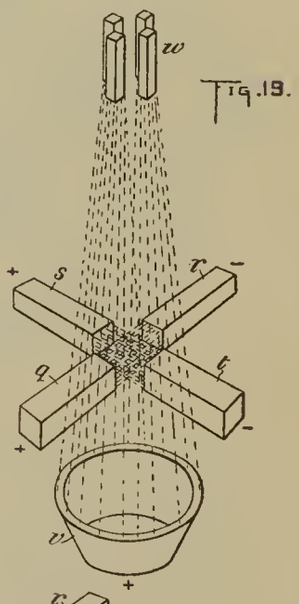
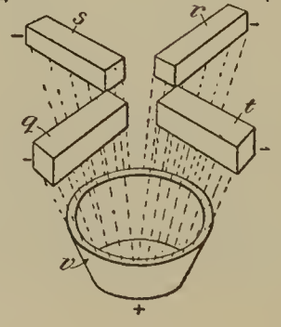


FIG. 20



chamber "w" into which is fed coarse ore, and if desired, fluxes. Outside of this again are shown the chambers "k" and "y" into which fine ore is fed, also fluxes if desired.

Baffle plates "p" are interposed in the walls that form the chamber "k-y," preferably of triangular shape, leaving a space below them so that carbonic oxide gases can pass from the interior to an outer chamber and in the passage come in contact with the descending fine ore, reducing more or less of it to the state of metal. More or less also of this ore in chamber "k-y" falls through openings under the baffle plates, and mingles with the coarse ore in the sections w. This carries radiated heat absorbed towards the interior, while its place is taken by cooler ore from above.

Electric motors are shown at X on the outer periphery of the furnace driving small fans which return the carbonic acid gas to the interior of the furnace, where it may be again reduced to the state of carbonic oxide, and made capable of reducing more ore to the metallic state.

Fig. 1 shows the hearth of the furnace, whose lining consists of carbon electrodes capable of being placed either in circuit with electrodes at the top of the furnace, or with the main electrodes at will. At one side is shown a spout connected with a taphole for the removal of the molten metal, and in dotted lines directly in front at the back of the structure, the tophole for the removal of slag. It is quite likely that the hip, or enlarged bottom (*Fig. 1,* bi-sulphide of carbon furnace) would be the better form for a furnace for this work. It is not necessary that the broken carbons should travel the whole distance from the top of the furnace to the electrode, and the hip would offer better facilities for the renewal of the electrodes. The screws for forcing the ore forward are shown in Fig. 2. These screws work in the outer and cooler portions of the fine ore, and are not intended to be subjected to any considerbale heat. They are hollow, as shown at H, and may be connected with a blast of air when desired to change the working conditions of the furnace; for example, to oxidize the charge to make it less conductive, or to burn out carbon when in excess. Fig. 2 also shows the column of charcoal maintaining itself down through the center of the shaft. Also the

demarkation of the coarse and fine ore in their progress. Fig. 6 shows how the different columns of coarse and fine ore and charcoal may be differentiated in their start from the top of the furnace. It is hoped by this means to impede the matting together of the charge and restrain the tendency to hang up. Fig. 7 shows the relative relation of the feeding screws to the main electrodes, in looking down upon them. By comparing Fig. 1 and 2 it will be seen that the screws are at a higher level than the electrodes. Fig. 2 also shows the outer column of ore which absorbs the last portions of radiant heat to be later or lower down pressed into the heat zone by screws provided for that purpose. It also shows the circulation of the gases through the different columns of ore by means of which it becomes reduced before reaching the zone of more intense heat. Fig. 4 shows the arrangement of hoppers and chargers on the top for feeding materials into the furnace.

Details of electrode and other constructions are shown in Figs. 8 to 20.

DISCUSSION.

Mr. Pedro G. Salom: I would ask Mr. Taylor if he has made any measurements of the amount of current in kw. hours per ton of iron?

Mr. Taylor: Mr. Hering made some calculations along that line, but they are not definitely in my mind at this time; I have the data from Mr. Hering for some of these. It is a matter of surprise how little electric current is required for these transformations. It is also interesting to note how small the quantity of carbon is which is required for the reaction. When you dispense with the blast passing through a blast furnace, you dispense with several things—you dispense with a large quantity of nitrogen and moisture going into the furnace, a large quantity of carbon otherwise going into the furnace, and consequently you have reduced to a simple proposition a great many other things.

I did not refer in my paper to the taking out of gases. Provision is made for that, the same as in the case of the bisulphide

of carbon furnace. This is a closed furnace, and the gases can be taken out and used in a gas engine, or in any other way that is desirable.

THE PRESIDENT: Perhaps Mr. Hering could give us an approximate idea, without going into many details, of what the current requirement is.

MR. CARL HERING: I do not remember the figures; it is some time ago since the calculation was made. Such calculations are, of course, at best only approximations, but I might call attention here to the fact that it depends very largely on whether the escaping gases are CO or CO_2. If the escaping gases can be made to be CO_2, the energy efficiency will, of course, be very much greater. CO_2 changes to CO at high temperatures in the presence of an excess of carbon, thereby greatly reducing the available energy.

PROF. J. W. RICHARDS: I sympathize very much with these attempts to construct an electric shaft furnace for the production of pig iron, and I think it will be only a very few years before there will be many of them in operation, particularly in such places as Norway, which has the advantages of pure ore and very cheap power.

However, I would criticise the structure which Mr. Taylor has proposed, particularly in respect to the feeding of the ore separately from the fuel, and keeping them separate as they go down in the furnace. It is a wrong practice in the working of a shaft furnace to keep the ore by itself and the fuel by itself. The fine ore, particularly, packs, and the reducing gases cannot pass through it. Therefore, the other point which Mr. Hering has mentioned, namely, the possibility of utilizing the CO and having a high percentage of CO_2 in the gases, can be attained only imperfectly with such an arrangement. I should say that the furnace is not adapted to securing a high efficiency of consumption of carbon, or of the heat of oxidation of the carbon.

Furthermore, if the fine ore is intended as a blanket for the sides of the furnace in order to bring back the escaping heat into the furnace, it will necessarily follow that the screws, operating in the fine ore at the lower part of their travel, must be in hot material, otherwise the ore will not have performed its func-

tion of intercepting the escaping heat. In hot material, I believe their working would be difficult, if not impracticable. These are a few of the points which strike me in regard to the construction. I may be a false prophet, but these are points of anticipated difficulty which experience in working other shaft furnaces brings to my mind.

In connection with the name of this furnace, I would ask all those present to use their influence against calling it the "electric blast furnace." In Germany they call it "elektrische hoch-ofen," which is quite natural, and in France they properly call it "haut-fourneau electrique," but if we call it the electrical blast furnace it will be giving it a misnomer. Let us call it the electrical shaft furnace, and then we will not be committing a crime upon the English language.

MR. C. A. HANSEN: I think it is due as a courtesy to Dr. Haanel to use the term "shaft furnace." Dr. Haanel has contributed so largely to the literature of the shaft furnace that I think the suggestion of Dr. Richards is well taken.

MR. F. A. LIDBURY: I do not quite understand whether the particular form of construction which is here shown is intended in any degree to get a better chemical efficiency out of the carbon; that is, with reference to that which is obtained in other forms of electric furnaces. I should like to know whether that is one of the objects of the novel design, or whether the objects are merely those of heat insulation, better heat efficiency and better operation of the furnaces. If it is expected that a better chemical efficiency is to be obtained from the carbon by this construction, I should like to know on what grounds that is anticipated.

MR. TAYLOR: The volume of charcoal, coming down, as I indicated, goes clear down through and between the electrodes. As I said before, it is not expected that these electrodes will be in the molten metal. The carbon or charcoal will pass down in between them. It is a matter of fact that in the blast furnace the principal reduction of the ore is not done by contact of the carbon and the ore coming into intimacy, but is done almost entirely by carbonic oxide. However, should any of the ore fail to be reduced in the separate state, as Dr. Richards refers to in his remarks, it could hardly fail to be reduced in its passage

between these electrodes and the charcoal; that is, as it goes through in drops (see M to 31, Fig. 1) it would be sure to be reduced, so that I cannot think but what the carbon would be fully and efficiently utilized in the furnace. I think the full utility of the carbon can be secured. There would be no objection to feeding some carbon with the ore, if it was found to be desirable, but I did not provide for that in the specification, for the reason that the small quantity of carbon that is required to reduce the ore in connection with the electric furnace, where you do not have a blast, may lead to an accumulation of the carbon in the interior of the furnace that we might like to avoid.

I should have said, and it is spoken of in the printed copy of the paper, that I have made the screws hollow with the idea of throwing a blast of air through them, by means of which any excess of carbon could be burned out or an oxidizing condition obtained which would reduce the conductivity of the furnace, so that I think we shall have no trouble about the efficient use of the carbon in the furnace.

PRESIDENT BAEKELAND: You speak about the reduction of the carbon dioxide. I suppose you mean to monoxide?

MR. TAYLOR: Yes, the carbon monoxide, that would circulate through the ore here, would be converted into carbonic acid, and then it may pass out into this chamber here and be driven by these fans from the motors through the ore again and into the interior, striking the carbon, and there be reduced to carbon monoxide. It may be driven back and forth several times. The fans are not set exactly opposite each other; if they were, they might baffle each other. They are set differently toward each other so as to produce a whirling circulation of the gases (see Figs. 3 and 15, X and O).

MR. LIDBURY: I am afraid I should have defined a little more clearly what I meant by the chemical efficiency of the carbon that is used. The information I wish is this, and the reason I ask it is because I did not understand the bearing of Dr. Hering's last remark: Is it anticipated that a larger amount of CO_2, relatively to the CO, will be obtained in the exit gases? Of course, the further the reaction between the CO and the iron oxide goes, the greater will be the chemical efficiency of the carbon. There

is, however, a ratio between the CO and the CO_2, which is governed by the conditions prevailing, *e. g.*, the temperature and the nature of the substances present, which fixes a limit beyond which no further reduction will take place at the expense of the CO. Is it anticipated that these conditions in this furnace will tend to a better utilization of the CO than in other furnaces, and, if so, on what grounds is this anticipation based? If it is not so claimed, of course, I only want a negative answer.

MR. TAYLOR: I think the answer, so far as we can answer the question from conditions that have not been tried, would be in this way: The carbonic oxide that would be last to come out of the charcoal that is heated in the interior, would make its way through the iron ore as the last passage, and would therefore have a tendency to form carbonic acid before it got to the outlet flues. By means of the blowers the gases circulate back and forth several times before their exit from the furnace, and the oxidation to carbonic acid and reduction to monoxide is repeated several times. This should mean a better use of the carbon. In reality the gases make their last passage through the ore and should be as far as possible converted into carbonic acid before they come out. I think in all furnace practice considerable carbonic oxide goes out with the gases, and it is probably not possible to make the conversion to carbonic acid perfectly complete in the effluent gases.

MR. C. A. HANSEN: I wish to say that I agree with Dr. Richards that the proper principle is to feed iron ore and carbon together in a reduction furnace. I have some data that bears in a roundabout way upon the subject. Some years ago we were interested in making ferro-silicon, and, among other experiments, tried to determine the effect of fine grinding of the charge upon the uniformity of yield and rate of reaction. The charge consisted of scrap iron, silica and carbon, a charge largely used in commercial manufacture. The apparatus used was the now well-known Arsem vacuum furnace[1] connected to a pump of constant displacement. When the charge is heated and CO is generated by the reaction, the manometer reading, when constant, will obviously be proportional, practically, to the reaction

[1] J. Am. Chem. Soc., 28, 922 (1906); Trans. Am. Electrochem. Soc., 9, 153.

rate. In one case the charge was broken to pass ⅛-inch square mesh, and the fines which passed through a 1/16-inch mesh were discarded. Manometer readings were taken at various temperatures. In another case the materials were all ground to extreme fineness, briquetted with a very dilute water-glass solution and baked to 960° C. A charge of the same weight as in the previous case was used, and manometer readings were again taken at various temperatures. The results in the two cases were as follows:

	CO Pressure readings	
Temperature °C	Coarse charge	Fine Charge
1400	0.4 mm	8 mm
1700	3. "	27 mm
1900	5. "	Off Scale > 60 mm
2100	9. "	

In other words, the reaction rate, which at the lower temperature depends solely upon contact between solids, is enormously increased by increasing the contact area. Making use of these results, I ran a briquetted charge in a furnace, the charge volume of which was scarcely over 1 cubic foot (magnetite, however, replacing the scrap iron) at a power input of 100 kw., turned out 50 per cent. alloy from this small furnace at slightly more than 2 kw. hours per pound, and practically all of the composition determined by the charge. The furnace did not get excessively hot, and there was very little fume. At this rate the commercial 1,000 kw. furnaces could absorb nearly 5,000 kw. by using finely-ground and briquetted charges. Obviously, the losses per unit output are enormously decreased by this procedure, and it becomes merely a case of balancing the cost of grinding and briquetting against the cost of power. In the case of ferro-silicon, so long as no one objects to the fumes, the balance is probably in favor of not grinding charge. I do firmly believe, however, that much is to be gained by bringing the solid reagents into contact with one another—the more intimately the better—in this case, in the case of iron ore reductions, and in all similar cases.

Mr. Taylor (*Communicated*): Answering further the questions of Mr. Lidbury: By reference to Figs. 2 and 3 respectively, please note the column of incandescent charcoal Fig. 2, so maintained by secondary electrodes shown at w and 9, Figs. 1, 2 and 3,

and in detail in Figs. 18 to 20, with their independent electric circuit. It is one of the offices of this incandescent column to reduce to monoxide any carbon dioxide forced into it by the blowers X and O. It is hoped that these alternate reactions will take place so frequently as to reduce to a minimum the amount of carbon required for the reduction of a given amount of ore. Regarding the suggestions of Dr. Richards and Mr. Hansen, the heat conditions are quite different from that of the blast furnace where the lining of the furnace suffers from the heat almost as much as the charge itself. In feeding around the periphery as proposed by me, in two columns—one outside of the other—it is certainly not expected that the outer of these columns will reach anywhere near a red heat before it has been pushed past the arches shown at l and k Fig. 3. Yet it should return to the furnace large amounts of heat, even if it should in a measure fall short of the good results of the carbon bisulphide furnace, on the outside of any part of which the hand can be placed without discomfort.

Certain it is if the circulation can be maintained like results may be expected. It will not be hot enough outside of the arches to cause the ore or metal reduced to stick. Inside the arches, where the temperature will be higher, one column will not be able to join the other (referring to the four main divisions) and the pressure from the screws and the descent of the materials determine the passage of the charge into the heat zone of the furnace.

Quite likely there will be some matting together of the materials just before the melting zone is reached, but the pressure of the ore pushed by the screws is designed to prevent its remaining in that position. The screws are expected to work exclusively in the outer column of the descending ore, which pushes forward the inner column. This insures the screws working in comparatively cool ore. (See Fig. 2). Reciprocating pushers can be substituted for the screws if desired.

Moreover it must not be overlooked that this furnace is not designed for small scale work; there is therefore plenty of room for the free movement of all the material.

A paper read at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30 1909; President L. H. Baekeland in the Chair.

PHYSICAL PROPERTIES OF IRON-COPPER ALLOYS.

By Charles F. Burgess and James Aston.

The influence of copper on iron and steel has for long been a subject of controversy and contradiction. It is only comparatively recently that the effect has been investigated systematically and some of the doubt eliminated.

The prevalence of appreciable quantities of copper in the ores of certain districts makes important a knowledge of its effect, detrimental or otherwise, when present in small amounts. Also, the intimate relation of copper, nickel and iron, and the beneficial effect of the addition of nickel to iron and steel, creates an interest in the influence of larger percentages.

The older opinion was that copper was deleterious; that its chief effect was analogous to that of sulphur in that it rendered iron red short and destroyed its welding power. This view was taken by many eminent metallurgists, some even contending that 0.5 per cent. makes steel worthless. Others claimed that the influence was greatly exaggerated, and instances were cited where rolled sections of 0.50 to 0.75 per cent. of copper did not display red shortness.

Within recent years more systematic investigations have been made to determine the effect of additions of varying percentages of copper to iron and steel.

Ball and Wingham[1] tested four steels with from 0.85 to 7.17 per cent. copper and a carbon content varying from 0.10 per cent. in the former to 0.71 per cent. in the latter, and noted an increase of tensile strength and a decrease of ductility with the increase of copper. The bar with 7.17 per cent. copper was red short. Their conclusions were that the principal effect of copper was to make

[1] Ir. & St. Inst., 1889, No. 1.

steel hard and that copper within reasonable limits did not materially affect the mechanical properties.

A. L. Colby[2] says that small percentages of copper have no deleterious effect upon the physical properties of steel. He cites various steels of about 0.5 per cent. copper in shafts and gun tubes, which met the requirements of the U. S. Navy. It was also used in ship plates, passing the usual tests required of carbon steels. These steels welded successfully and flanged cold and there was no red shortness in bars or rails with a copper content of 0.39 to 0.49 per cent.

W. Lipin[3] states that iron with a copper content up to 3 per cent. is readily worked but that there is red shortness at 4.7 per cent. copper. Between 7 to 10 per cent. Cu the material cracked badly, and fell to pieces under the hammer. With an increase of copper up to 3 per cent., the tensile strength increased from 26 tons per square inch to 46 tons per square inch, with a decrease of elongation from 27.8 per cent. to 13.3 per cent. He also noted that with an increase of carbon in the steel the maximum percentages of copper must be decreased; also that copper does not affect the welding.

Stead[4] made a very thorough investigation of the influence of copper in steel rails and plates, since, he claimed, the general belief that copper was deleterious was an unjustified prejudice. His article goes fully into the previous work and impressions. He finds that copper between 0.5 and 1.5 per cent. is not detrimental either hot or cold; that 2 per cent. copper makes steel more liable to show the effects of overheating; that small quantities raise the tenacity and the elastic limit and reduce the elongation, but not greatly, however, for small percentages of copper; also that there is no great liability to fracture by shock.

Papers of Stead and Wigham,[5] and of Wigham,[6] deal with the effect of copper in cold wire drawing, and come to the conclusion that copper up to 0.25 per cent. is no disadvantage in the manufacture of the best classes of steel wire.

Wigham, in the last article mentioned above, brings out the

[2] Iron Age, Nov. 30, 1899.
[3] Stahl u. Eisen, Vol. 20.
[4] Ir. & St. Inst., 1901, No. 1.
[5] Ir. & St. Inst., 1909, No. 2.
[6] Ir. & St. Inst., 1906, No. 1.

important point that Professor Turner, in the discussion of the former article of Stead and Wigham, says that where copper is present in ores it is found in association with sulphur. Consequently discrepancies in the earlier tests may be due to this sulphur and not to the direct effect of the copper. This fact, together with that of the increase of red shortness with an increase of carbon, may explain the differences of opinion resulting from the earlier observations.

All of the above research was to excuse the presence of the copper; to break down a seeming prejudice. An investigation with a different object is that of Pierre Breuil, the results of which were given in a paper before the Iron and Steel Institute.[7] His object was to see if there was a beneficial effect due to the addition of copper, and was suggested by the favorable influence of copper on steels for railway axles, which was noticed on some of the French railroads. It is a very extended research, and, as the author says, is a supplement (very much elaborated) to Stead's work, in that the results agree. Being the most exhaustive and latest contribution to our knowledge of copper steels, we shall have occasion to refer to the results frequently throughout this discussion.

Breuil's work was carried out on four series of steels, mild steel (C 0.10—0.17 per cent.); semi-mild steel (C 0.28—0.41 per cent.), and hard steel (C 0.56—0.79 per cent.), and a final series with about 1 per cent. of carbon.

In view of the evidence that the influence of copper varies with the amounts of carbon and of sulphur present in the steel, and very likely also with the other impurities met with in commercial materials, it is of interest to record the results of tests where these elements are a minimum. This study is a part of an extended investigation carried on in the Chemical Engineering Laboratories of the University of Wisconsin under a grant of the Carnegie Institution of Washington, on electrolytic iron and its alloys. Much of the data and results here enumerated were obtained by Messrs. Watts, Kowalke and Thickens.

The primary element in the production of these alloys is iron of high purity, obtained by double electro-deposition. The nature of the material is given in the following table:

[7] Ir. & St. Inst., 1907, No. 2.

Per cent.	Anode	Double Refined	Electro Fused and Forged
C	0.260	0.012	0.047
S	0.007	None	0.005
Si	0.019	0.013	0.062
P	0.007	0.004	0.016
Mn	0.021	None	None
Iron	(difference)	99.971	

As will be seen from this table, the iron content is 99.97 per cent. The copper used is also of high purity, being obtained by electrodeposition.

The charges were made up in dense electrically shrunk magnesia crucibles, each enclosed in a graphite container. The crucible was covered with a magnesia lid and was buried in an electric resistor furnace. The charges in the crucible were brought to fusion from the cold mixture, held at a high temperature for some time and allowed to cool in the furnace. There was no agitation of the charge and the resultant alloy was due entirely to diffusion. The ingots weighing about 500 grams (1 lb.) were forged into rods about $\frac{5}{8}$″ in diameter by 18″ long, under the steam hammer, and were then turned or ground into the finished test bars.

In spite of precautions to prevent contamination of the charge, some carbon was absorbed. Evidence is that it was set free by the equilibrium reaction, $Fe + CO = FeO + C$. The amount is small, however, and numerous analyses show that it may be taken as well under 0.10 per cent. The other impurities, such as manganese, silicon and sulphur, are negligible. In the tests here enumerated therefore, the effect may be considered as due to the added element only.

Forging.

A wide range of alloys of varying copper content was made. The observations made in the forging are as follows: Alloys up to 2 per cent. of copper forge well at low heats. Those from 2 per cent. to 7 per cent. will not forge at a low heat, and rather poorly at white heat, the ease of workability varying inversely as the percentage of copper. From 7 per cent. to 75-80 per cent. the alloys may be classed as non-forgeable. Between 80 and 100 per cent. they will forge at a fair red heat but not at a normal forging heat for iron.

In the earlier work alloys above 5 per cent. could not be forged. However, in later tests, in the trial of many alloys of 5 to 10 per cent. of copper at all heats, it was found that with care and a high heat (welding) a 7 per cent. bar could be forged, or even rarely, an 8 per cent.

The lower percentages (below 5) will weld easily in forging, and some bars of 7 per cent. copper were welded while forging. All alloys up to 7 per cent. copper could be welded in an electric welder.

Segregation.

The forgeable samples between 0 and 8 per cent. of copper and a few of the alloys of high copper and low iron were made into test bars for investigation of the tensile strength. Analyses were not made upon all bars; rather, random samples of the entire range of composition were sent to Booth, Garrett & Blair, of Philadelphia, whose valued assistance in this investigation is acknowledged. Their report is given below:

Analyses.

Bar	Cu Added	Analysis
147B	0.1	0.089
158A	0.2	0.202
158B	0.4	0.422
158C	0.6	0.592
158D	0.8	0.804
147H	1.0	1.006
147J	1.5	1.510
158G	2.0	2.005
158I	4.0	3.990
158J	5.0	5.070
158K	6.0	6.160
147U	7.0	7.050
86F	95.0	94.340

As will be seen from the tables, the agreement between the added and the actual copper content is exceedingly close. The analytical samples were taken from the turnings obtained in the machining of the test bars after the forging of the ingots. This close agreement after such a method of selection would indicate

an entire absence of segregation; also that the materials alloy very well up to a copper content of 7 per cent. This is in accord with the work of the earlier investigators. In view of this close agreement it may be assumed that the actual copper content of the alloys is the same as the added amount.

Hardness.

The hardness tests are merely qualitative, being observations made in the machining, sawing and filing. In machining, the alloys with low percentages of copper worked very easily. The hardness increased with the increase of copper until at 5 to 7 per cent., while it was still possible to turn in the lathe, it was with some difficulty. There were the same evidences in the sawing and filing tests, the high copper being classed as extremely hard to saw.

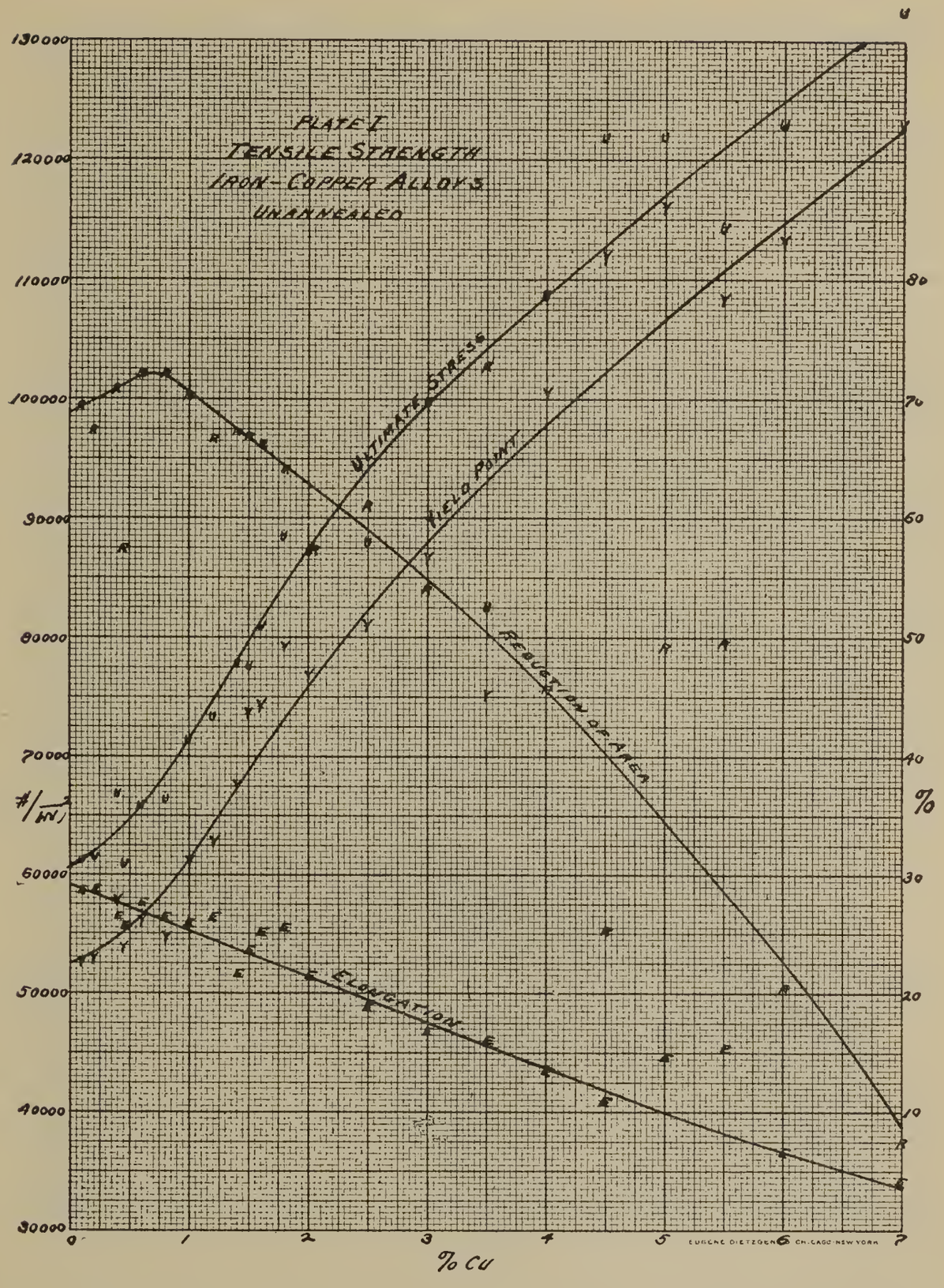
Breuil's investigations show that the hardness does not result through a lowering of the points of transition, thus leaving the steels in a martensitic state, as might be supposed. The structure is a fine pearlite, with a fibrous cementite which is liberated with increasing copper, and this explains the increasing hardness.

Wedding[8] says that copper and sulphide of copper prevent the formation of pearlite and promote the formation of crystals of cementite. It is due to this fact that there is greater hardness of iron when copper and sulphur are present. These explanations seem hardly sufficient to account for the hardening which we have observed without the presence of either copper or sulphur and we expect to make a metallographic study with the hope of throwing a little light upon the subject.

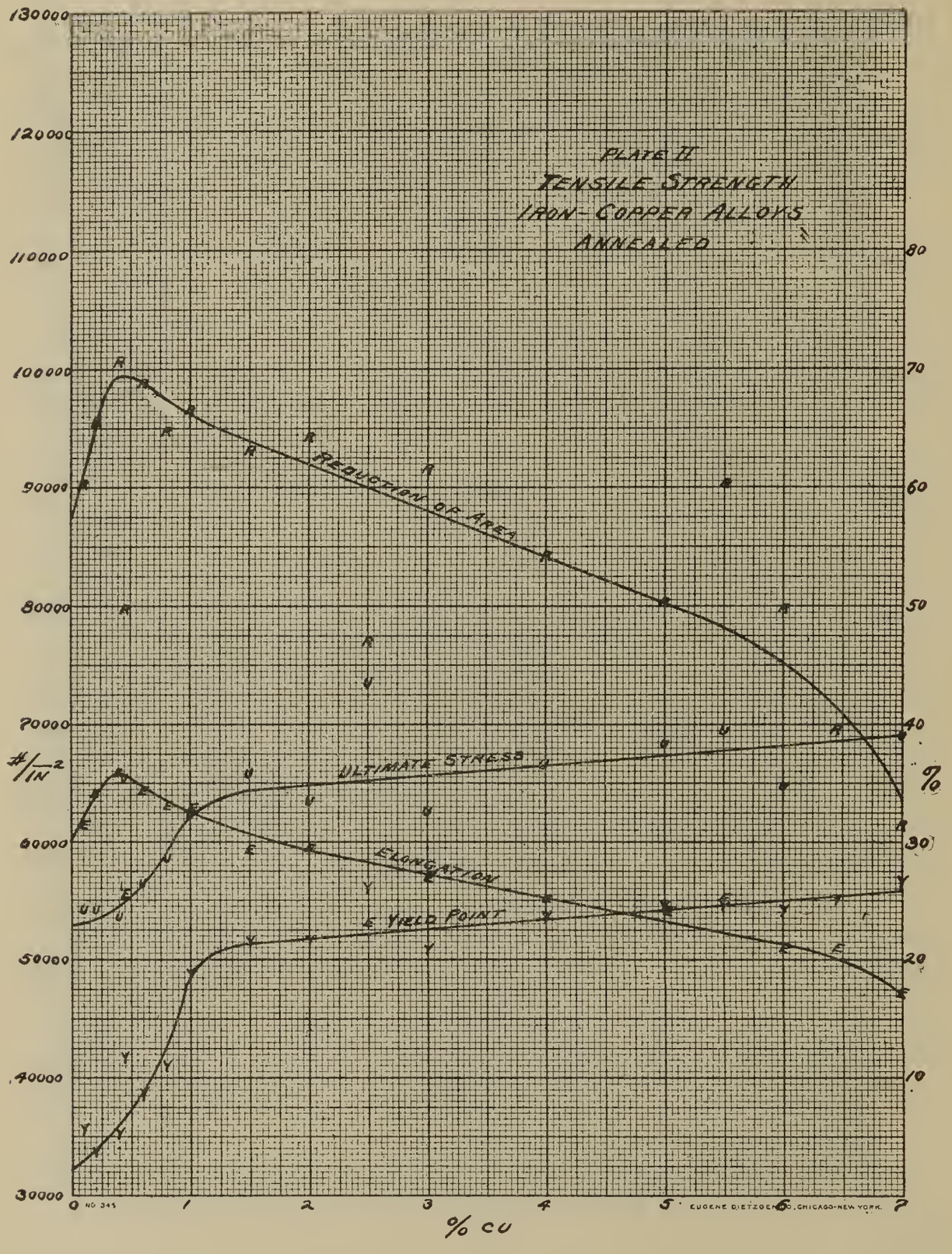
Tensile Strength Tests.

Of the bars cut from each rod for the tensile tests, an equal number were reserved for tests as forged and after annealing. In annealing, the samples were packed in a magnesia lined graphite box and were heated in an electric resistor furnace. The temperature was brought up to 900° C and held at this point for several hours, and several hours more were needed for the slow cooling in the furnace. The test samples were approximately

[8] Stahl u. Eisen, December, 1906.



from 0.3 to 0.4 inches in diameter, with a free length of 2 inches. The diameter used in the calculations is the mean of several readings with micrometer calipers. The tests were made on a Riehle 100,000-lb. power-driven machine. The load was applied very slowly and the beam kept in balance. The yield point was obtained with dividers, and was taken as that point at which a



perceptible permanent stretch had taken place. This was checked by the drop of the beam.

The summary of the results of these tests is given in the Tables I and II and in Plates I and II. The detailed data are not given, also no account is taken of bars which showed abnormal results, due to flaws, etc.

TABLE I.

Tensile Strength—Iron-Copper Alloys—Unannealed.

Per. Cent. Cu.	No. of Samples	Yield Point Lbs./in²			Ultimate Stress Lbs./in².			Elongation %/2″			Reduction of Area Per Cent.		
		Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.
0.1	3	56,600	45,400	52,580	64 000	60 300	61,180	30.0	27.5	28 5	70.9	66.7	69.2
0.2	5	55.600	50,700	52 930	63.750	60,000	61 470	36.5	24 5	28.7	73 2	56.9	67.2
0.4	2	59,700	56,100	57.900	67 250	66.400	66.825	29.5	23.0	26.2	72 1	69.4	70.7
0.45	2	54,800	53,100	53 950	62 700	58.800	60,750	27.0	24 0	25.5	67 7	47.2	57.4
0.6	2	57,750	54,600	56.170	67,000	64,750	65 875	27.5	27.5	27.5	72.8	71.5	72.1
0.8	2	55,500	53,800	54.650	67 500	65,100	66.300	27.0	25.5	26 2	72.5	71.7	72.1
1.0	5	63,300	56,800	61,250	70 800	68 300	71,380	28.0	21.0	25.8	72.0	66.8	70.3
1.2	2	66,300	59 500	62,900	74,600	72,300	73.400	28.0	24.5	26 2	68.8	64.5	66.6
1.4	2	68,100	67,300	67 700	78.600	76,600	77 600	25.0	18.0	21.5	70.0	64.5	67.2
1.5	3	77,250	70,000	73,920	78,800	76.200	77,300	26.5	20.5	23.5	68.6	64.5	66.9
1.6	2	75,100	73,300	74.200	82.750	79.100	80,925	25.0	25 0	25.0	66.6	66.2	66.4
1.8	2	81,100	77,600	79 350	91.000	86,000	88.500	27.0	24.0	25 5	66.3	61.8	64.0
2.0	3	78,800	73,400	76.930	89 500	82,250	87.010	23.5	17.5	21.2	59.5	54.5	57.0
2.5	2	81,100	81,000	81.050	89 400	86.400	87.900	19.0	18 5	18.7	62.8	59.1	61.0
3.0	6	95,100	73,750	86,810	105.000	83,750	99,640	24.0	12.5	16.7	65.6	36.1	53.8
3.5	1			75,400			82 300			16.0			72.7
4.0	5	111,100	92,700	100 560	120.700	99,200	108.640	17.5	8 0	13.5	52.3	24.8	45.6
4.5	2	117,800	106,400	112,100	127 900	115.500	121,700	17 0	4.5	10.8	47.2	3.9	25.1
5.0	2	119,700	113.000	116,350	123 800	120.000	121,900	15.0	14 0	14.5	50.1	48.3	49.2
5.5	2	114,700	102.300	108,500	122 800	105,200	114,000	15.5	15.0	15.2	53.8	46.6	49.7
6.0	3	124,200	103 000	113.400	136.700	107,500	122,900	9.0	4.5	6.5	36.5	4.1	20.3
7.0	2	128.800	117 000	122,900	133 800	131,000	132.400	6.0	2.0	4.0	12.9	1.7	7.3
8.0	2	146,800	133.500	135,100	160,000	137.500	148.750			0			0

TABLE II.

Tensile Strength—Iron-Copper Alloys—Annealed.

Per Cent. Cu.	No. of Samples	Yield Point Lbs./in²			Ultimate Stress Lbs./in²			Elongation Per Cent./2″			Reduction of Area Per Cent.		
		Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.
0.1	2	40,750	34,400	35,570	56,500	51,600	54,050	35.0	28.0	31.5	62.2	58.3	60.2
0.2	4	36,850	31,800	33,660	56,000	52,700	54,020	36.0	29.5	34.1	67.7	62.7	65.6
0.4	2	35,700	35,100	35,400	53,700	53,100	53,400	37.5	34.0	35.7	72.1	69.3	70.7
0.45	2	42,250	41,250	41,750	66,300	64,600	65,400	26.0	25.0	25.5	53.6	46.0	49.8
0.6	2	38,400	38 300	38,850	56,600	56,500	56,550	36.5	32.0	34.2	69.0	68.8	68.9
0.8	2	41,300	40,700	41,000	59,750	57,400	58,570	34.0	32.0	33.0	65.1	64.7	64.9
1.0	4	54,800	46,400	48,920	63,900	61,100	62,350	34.0	30.0	32.6	68.3	66.1	66.8
1.5	6	58,100	48,100	51,570	70,500	62,100	65 720	33.5	22.0	29.2	65.1	59.3	63.1
2.0	6	54,300	49,400	51,620	65,100	60,500	63,200	31.5	27.5	29.5	68.1	62.0	64.4
2.5	1			56,200			73,500			23.0			47.3
3.0	7	53,800	48,700	50,890	67,600	59,000	62,360	33.5	10.5	26.9	69.1	46.3	61.7
4.0	5	57,100	49,600	53,570	70,900	62,700	66.540	28.0	22.0	24.8	65.3	39.3	54.2
4.0	4	57,600	50,700	54,460	74,700	62,400	68,250	29.0	20.5	24.2	62.9	29.1	50.3
5.5	1			54,800			69,300			25.0			60.4
6.0	3	55 500	52,750	54,420	65,600	62,500	64,530	24.5	18.0	20.8	59.0	39.2	49.9
6.45	2	55,750	54,600	55 170	69,700	69,200	69,450	25.5	16.5	21.0	48.1	30.5	39.3
7.0	2	57,150	56,750	56,950	70,900	67,900	69,400	20.0	14.0	17.0	40.8	21.7	31.2
7.5	1			60,200			79,000			16.5			34.2

The results enumerated are the yield point, the ultimate strength, the percentage of elongation in 2 inches and the percentage of reduction of area at fracture. In order to show the consistency of the data there is also given the maximum and minimum values for each of the above items, together with the average of these items for the number of samples enumerated in a separate column. The plates are plotted from the averages of the tables and it is not to be expected, considering the many variables which might arise, in tensile strength tests, that any series will give a simple curve. It is merely hoped to show the tendency of the increasing percentages of copper. The values of the yield point, ultimate strength, elongation and reduction of area are designed as points used in plotting the curves by the appropriate letters Y, U, E and R.

Unannealed.

From the curves of Table I, it will be noted that the rise in the ultimate strength and the elastic limit is almost a linear function of the percentage of copper. The ultimate strength increases from 61,180 lbs. per sq. in. at 0.1 per cent. copper to 132,400 lbs. at 7 per cent. The yield point rises from 52,580 to 122,900. There is a corresponding fall in the elongation from 28.5 per cent. at 0.1 per cent. copper to 4 per cent. at 7 per cent. copper. The reduction of area increases slightly from 69.2 per cent. at 0.1 Cu to 72.1 per cent. at 0.6 and 0.8 per cent. Cu; from this point it falls to 7.3 per cent. at 7 per cent. of copper.

From Table I it will be noted that the ratio of maximum and minimum values to the average is good, except in certain instances which are no doubt somewhat abnormal, but not so much so as to be rejected from the tables. Also, where there are a sufficient number of samples for a good average, the results fall well in line. The greatest variation is at 3.5 per cent. of copper, but here there is only one bar, which is clearly abnormal, with its low ultimate strength and elastic limit and its high reduction of area. The greatest variations from the curve lie above 4 per cent. of copper, and this is to be expected, especially in the unannealed samples, since here we are almost beyond the limit of satisfactory workability.

The results of these tests indicate a high tensile strength which

increases with the percentage of copper. The alloys are rather brittle as indicated by the elongation, and the reduction of area. Also, the elastic ratio (ratio of the elastic limit to the ultimate strength) is large, varying from 0.86 at 0.1 per cent. copper to 0.93 at 7 per cent. Cu.

Annealed.

The results of annealing are given in Table II, and Plate II, and the effect is very marked, especially for the higher percentages of copper. There is greater consistency in the results, as is to be expected, and the points fall fairly well in line on the curves.

The greatest deviation from the curves is at 2.5 per cent. and 5.5 per cent. of copper, where there was only one bar of each for the test. The maximum and minimum figures of the table are not widely different.

Up to 1 per cent. copper there is a marked increase of the elastic limit and the ultimate strength with the additions of copper and no falling off in the elongation or reduction. In fact, the latter increase to a maximum at 0.4 per cent. of copper. Beyond one per cent. the curves bend sharply and become more nearly horizontal, being a linear function of the percentage of copper. The elongation and reduction of area fall off in accordance.

Between 1.5 per cent. and 7 per cent. of copper the elastic limit increases from 51,570 to 56,950 pounds, the ultimate from 65,720 to 67,900 pounds, while there is a decrease in the elongation and the reduction of area. Between 0.1 and 1.5 per cent. of copper the elastic limit rises from 35,570 to 51,570 pounds; the ultimate strength from 54,050 to 65,720 pounds; while the elongation and the reduction of area remain about the same. Breuil mentions the 4 per cent. alloy as worthy of further study, and this is no doubt true, since he observes very high values, as indicated by the following table, where the results are given for his mild steel series (C = 0.10 to 0.17 per cent), as rolled, and as annealed at 900° C.:

Tensile Strength—Breuil.

UNANNEALED

Per Cent. Cu.	Yield Point Lbs. /sq. in.	Ultimate Stress Lbs. /sq. in.	Elongation Per Cent.	Reduction Area Per Cent.
0	. . . .	. .	. . .	. .
0.5	55,000	66,800	25.5	66
1	58,900	70,200	26.5	60
2	67,300	88,700	16.0	58
4	97,600	109,500	13.0	46

ANNEALED

0	35,800	54,900	30.3	63
0.5	38,200	59,000	28.0	60
1	54,800	69,500	26.0	57
2	58,700	70,300	25.0	58
4	65,000	71,500	22.0	63

A comparison of these figures with the results of our researches shows a great similarity, and in plotting the figures there is a very close agreement. In his 4 per cent. alloy as unannealed, to which Breuil calls particular attention, there is an elastic limit of 97,600, an ultimate strength of 109,500, an elongation of 13 per cent. and a reduction of area of 46 per cent. In our tests the results for corresponding material are—elastic limit, 100,560; ultimate strength, 108,640; elongation, 13.5 per cent. and reduction of area 45.6 per cent. For the annealed sample, Breuil gives the elastic limit, 65,000 pounds; ultimate strength, 71,500; elongation, 22 per cent; reduction of area, 63 per cent. Our results are: Elastic limit, 53,750 pounds; ultimate strength, 66,540 pounds; elongation, 24.8 per cent.; reduction of area, 54.2 per cent. In our tests the annealing shows a greater effect, resulting in less strength with greater ductility; and the same is true in the comparison for all percentages of copper.

While Breuil lays particular stress on the 4 per cent. alloy, our results would seem to indicate the greatest value between 1 and 2 per cent., for the annealed samples. The very sharp rise of the elastic limit and the ultimate strength at this percentage gives values but very little less than those for higher copper content; also there is a removal from the region where the forging

and welding properties are poorer with consequent uncertainties of result. Likewise, between 1 and 2 per cent. the elastic limit and the reduction of area are very high.

It would hardly be advisable to work with 1 per cent. of copper, since this is on the edge of the limit, where there is liable to be a fall to the lower values obtained with lower percentages of copper. At 1.5 per cent. we are far enough removed so that the slight differences of composition to be met in practice would not bring the material into a dangerous region. Also, the elastic ratio of 0.78 is slightly lower than for the 4 per cent. alloy, where it is 0.81.

We would have considerable hesitation in bringing forward this point without a vast number of tests for confirmation, but for the fact that an observation of Breuil's results indicates the same condition. He has perhaps noticed this fact, but makes no mention of it in his article. As will be seen from the table of his results which is given above, there is a sharp increase between 0.5 per cent. and 1 per cent. of copper, where the elastic limits are 38,200 and 54,800, respectively, and the ultimate strengths 59,000 and 69,500. Again, we note but little change in the elongation and reduction of area. Between 0 and 0.5 per cent., and between 1 per cent. and 2 per cent., the change is not marked. The fact that two independent investigations give this result, seems fairly conclusive of the effect.

Another fact to be noted in our tests is the marked difference between the unannealed and the annealed samples. It would indicate that there is a possible intermediate heat treatment which, not being so drastic in its effect as our long annealing, would give intermediate values, and thus approximate the nickel steels. This condition might be reached in commercial rolling, where heavier masses of metal leave the material in a condition analogous to that resulting from a partial anneal.

To compare the copper-iron wth the nickel alloys we may quote Waddell's extensive investigations.[9] In considering the use of nickel steel for bridge building, he mentions a steel with about 3.5 per cent. nickel, carbon 0.38 per cent., the values for which may be taken as—elastic limit, 60,000 lbs. per sq. in.; ultimate, 105,000 lbs. per sq. in.; elastic ratio from 0.55 to 0.60. These

[9] Proc. Am. Inst. Civ. Engrs., Sept., 1908.

values are for sections as rolled without heat treatment and working at a somewhat low ductility, as shown by the figures 15 per cent. for nickel steel, 27 per cent. for carbon steel (in 8 inches) and a reduction of area of 41 per cent. for nickel, compared to 55 per cent. for the carbon steels. With the carbon reduced to 0.15, the values become— elastic limit, 45,000; ultimate, 70,000; elongation, 25; and for (C= 0.45) the elastic limit is 65,000, the ultimate 115,000 and the elongation 12 per cent.

These values are higher than ours for the copper iron alloys but are not in direct comparison. Our own figures for a nickel-iron alloy made under the same conditions as the copper-iron series are given in the following table, where the 4 per cent. nickel is compared with the 1.5 per cent. and 4 per cent. copper alloys. There is also given Hadfield's test of an alloy of 3.82 per cent. nickel and carbon = 0.19 per cent.[10]

UNANNEALED

	Elastic Limit	Ultimate Strength	Elastic Ratio	Elongation Per Cent.	Reduction Area Per Cent.
4.00 Ni.	67,000	76,000	0.88	28.5	68 6
1.50 Cu.	73,920	77,300	0.95	23.5	66.9
4.00 Cu.	105,560	108,640	0.97	13.5	45.6
3.82 Ni.	62,720	82,800	0.76	30.0	54.0

ANNEALED

4.00 Ni.	57,000	69,000	0.83	26.5	64.8
1.50 Cu.	51,570	65,720	0.79	29.2	63.1
4.00 Cu.	53,570	66,540	0.81	24.8	54.2
3.82 Ni.	56,000	73,920	0.76	35.0	55.0

The above values for the nickel alloys are less than are commonly assigned to commercial materials where the carbon is a factor. The results are very comparable to those of the copper-iron series made under identical conditions.

Waddell, in his paper, shows that there is an economic advantage in the use of the nickel steels in comparison with the carbon steels ordinarily used, since their increased cost is more than

[10] Inst. Civ. Engrs., March 28, 1899.

offset by the less weight required, due to their increased strength. Our results and comparisons would indicate that the copper-iron alloys are also worthy of consideration and might be comparable to the nickel steels in use, even if the strength should not reach such high values as those of the nickel. A 1.5 per cent. copper alloy is of promise, since the smaller percentage required and the lessened cost per pound of copper as compared with nickel would result in a lessened cost of construction, even if there is some increase in tonnage required because of the slight decreased strength per equal weight.

Chemical Engineering Laboratories,
University of Wisconsin.

DISCUSSION.

MR. CARL HERING: This paper is of great interest, and we are fortunate in having such papers at our meetings. In looking over the results of others, as well as those in this paper, it appears that there sometimes is a decided discrepancy between the chemical analyses and the physical properties, and it therefore occurred to me that the copper might perhaps act differently, depending upon the physical treatment which the iron received prior to the testing of the sample; for instance, the chilling, hammering, annealing, etc If so, it would explain why the chemical analysis is not a criterion of what the physical properties would be.

PROF. J. W. RICHARDS: I would like to say, speaking from the metallurgical side, that this paper will be received with great interest by the manufacturers of steel in the United States. I was told privately some years ago by the metallurgist of one of the large steel works, for my own private information, that a great deal of their steel had from 0.5 to 0.7 copper in it, and passed all the requirements of the United States Government, proving that it was not injurious to the steel, but rather beneficial. That was then private information. Prof. Burgess' paper makes it public information, and even goes further than the information that was then known. I think it is one of the

most important papers, in its commercial aspect, that has been brought before the Society.

Dr. Arthur H. Elliott: The addition of these metals to iron for certain purposes is extremely interesting, and the one that strikes me as most interesting is its effect on the corrosion of the metal, especially in the making of pipes. This question came up last year at our meeting, and there was a great deal of work done on it. I wonder if there has been anything done with these iron-copper alloys in that direction. It is extremely important, the corrosion of these alloys, because of the small quantities of metals in them.

Mr. Roger C. Aldrich: I should like to ask whether any experiments were made on the addition of copper to steel. I notice it was stated that the irons had practically no carbon in them, and if any such experiments were made, I would ask if they had the same effect of raising the ultimate strength and elastic limits on the steels as on the iron?

Prof. Burgess: We did not make a series of copper alloys with steel. We made enough accidentally to find that in the presence of carbon the copper acts in a way entirely different from that described in the paper. It makes it brittle and red-short, and it does not appear desirable to add copper to high carbon steel.

We are studying corrodibility of several hundred alloys which are exposed to various corrosive conditions, and it is too early to make reports; but I am inclined to think that copper-iron is going to be a rather corrodible metal. Unfortunately this is a factor which cannot be determined definitely until after several years have elapsed.

As to Mr. Hering's comment, I entirely agree with him that the physical working of the material, forging as well as the heat treatment of these alloys, has much to do with the strength, and I think that is shown by the relative positions of the curves shown on pages 247 and 248.

Dr. Elliott: I think that is very interesting, with regard to the corrosion of the metal, but I did not intend to draw Prof. Burgess's fire. I think that is a good program for another paper.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City. October 30, 1909; President L. H. Baekeland in the Chair.

ELECTRO-CEMENTIZING.

A PROCESS FOR SIMULTANEOUSLY ANNEALING, CLEANING AND ZINC-COATING WIRE.

By Alfred Sang.

Iron or steel wire for fencing, barb-wire and wire goods of various kinds, is usually zinc-coated by the following method: The wire is drawn successively thru,

First, a tubular annealing furnace of considerable length,

Second, a solution of hydrochloric acid which removes scale and the carbon left by the burning of traces of wire-drawing grease,

Third, chloride of zinc, or else a mixture of sal-ammoniac and glycerine floating on the surface of,

Fourth, a bath of molten zinc at a temperature of about 500° C. and

Fifth, wipers (made of asbestos pads prest together by vertical screws), and thence thru water to a take-up frame.

These five operations of annealing, cleaning, fluxing, coating and wiping must be controlled within definite limits; any single one of the many possible errors of manufacture will affect the quality of the product.

The drawbacks inherent to the hot process as applied to wire are five in number:

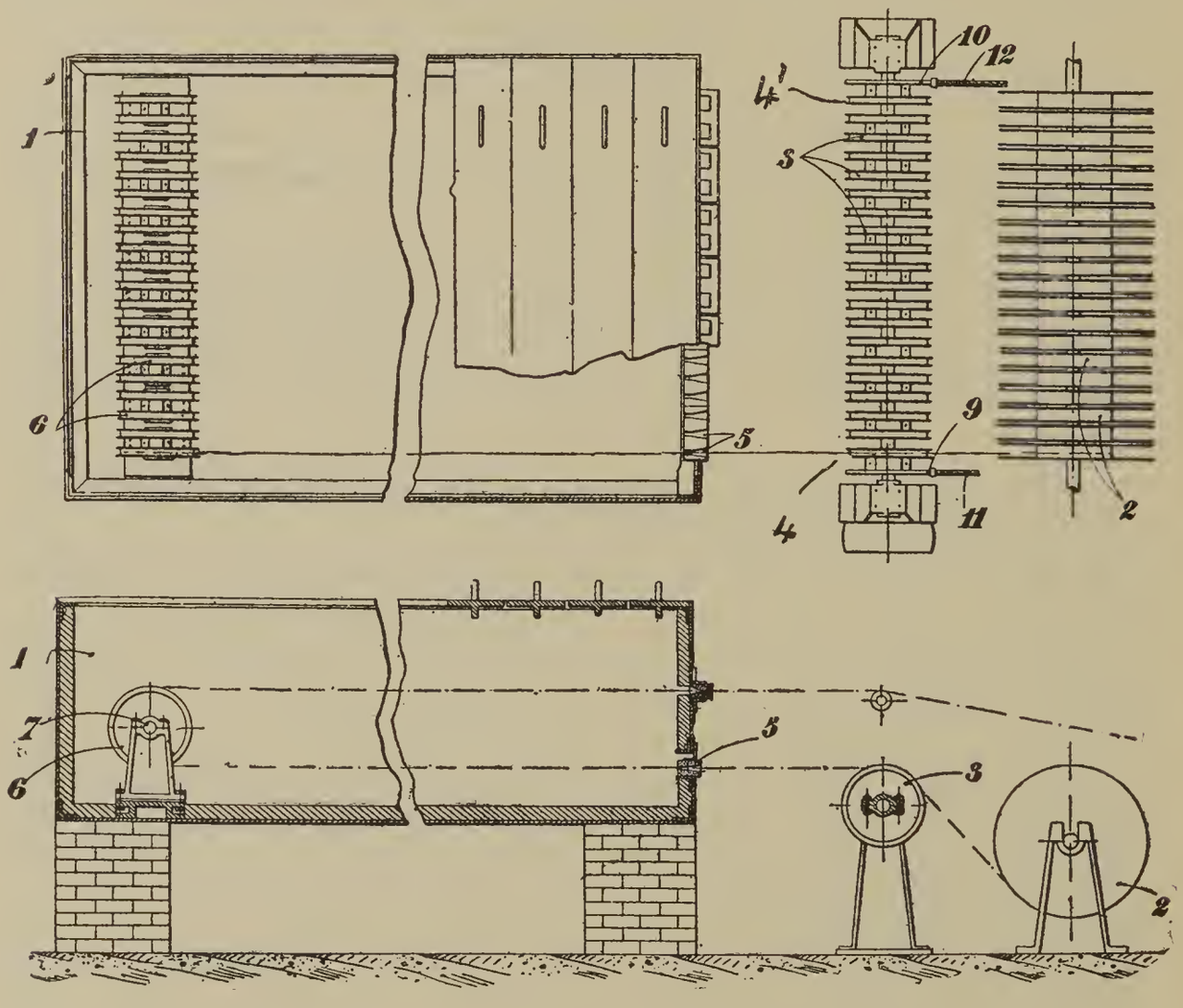
First, the annealing is imperfect;

Second, the acid bath causes the absorption of hydrogen, which not only hardens the surface, but promotes subsequent corrosion and prevents the intimate adherence or alloying of the zinc to the iron;

Third, the flux causes pockets to form between the zinc and the iron, in which traces of chlorides are imprisoned and assist corrosion; the wiping destroys the pockets themselves, leaving areas where the coating is practically non-adherent;

Fourth, the zinc deposited is high in iron and therefore more readily scaled off than pure zinc and sooner destroyed by moist air and other agencies;

Fifth, the wiping is difficult to control; if too close it will very often cause streakiness, the protection to the iron being that afforded by the amount of zinc at the thinnest spots; if the zinc is wiped too cold there occurs a transversal crackling not unlike the hair-cracks in a troweled surface.



The hot process is not a simple one and an expert wire galvanizer is a valuable specialist; it only appears simple and advantageous as compared to more recent processes, because there are two generations of experience back of it.

The interest recently shown by the United States Department of Agriculture, and the investigations which are being also carried out by leading wire manufacturers, indicate that there is a demand from farmers and others for something better than the present grade of galvanized wire. In the hope of helping

the question and of drawing attention to what is probably a new field of experiment, I submit the following process,* which has been tested in a modest way and, it is hoped, will soon be tried, for a verdict of life or death, on a small commercial scale.

The claims to attention possest by this process are its comparative simplicity, the fact that it lends itself to scientific control and the quality of the product. I seriously doubt if it can equal the low costs for hot galvanizing under conditions as they now exist, unless, perhaps, it be applied on a very large scale.

The principle involved in this new process, which I have called "electro-cementizing"—cementizing being the process of depositing metals by cementation, of which sherardizing is a form—is by no means new. We find in the Bulletin de la Société de Chimie et de Physique for the year 1865, page 374, that M. Dumas had found a means of zinc-coating iron by burying it in oxide of zinc and powdered charcoal and heating it to redness. Since then, many processes depending on the reduction of oxides or oxidized dusts in contact with the articles to be coated have been tried, usually with indifferent success.

The novelty of electro-cementizing consists in the method of heating by electricity, the heat being directly applied to the articles by using them as resistances. On the very face of it, it is only applicable in practice to articles of small cross-section, such as wires and strips, altho in theory it may be applicable to larger goods, and if carried out on a very important scale could undoubtedly be applied to thin sheets and tubes.

Referring to the diagram: the coating chamber or furnace (1) is built of iron plates and lined with refractory material as used in zinc smelting furnaces; it is filled from the top with a mixture of crusht coal and oxide of zinc. The wire comes off the reels (2) and passes over and around brass contact sheaves (3) which are equal in number to the strands of wire which the furnace will treat at one time, and are mounted tight on a common shaft from which they are insulated by non-conducting bushings. This shaft, by revolving, leads the wires and relieves the tension on them. The single sheaves at each end of the shaft (4, 4′) are connected thru the proper rheostat to the poles of a suitable transformer supplying alternating current at a low tension which may

* Patents allowed.

be varied within certain limits. The intermediate sheaves are paired by means of common hubs so that the current may pass freely from one to the other in each pair.

The wires now pass between the hinged aprons or flaps (5) and into the lower part of the furnace, then over another series of sheaves (6) inside and at the rear end of the furnace, these sheaves being similar in every respect to the double sheaves on the outside but staggered in relation to them, by which is meant, that the near sheave of each outside pair is in line with the far sheave of each inside pair; the wires then return thru the upper part of the furnace and thru a second set of hinged flaps, to the take-up frame which would, if shown in the drawing, stand to the right of the paying-out reels. The valuable idea of bringing the wire back thru the upper part of the furnace was suggested to me by Mr. Hadaway of the Westinghouse Electric and Mfg. Co.

The entrance and exit flaps will allow the passage of the loops which connect successive strands end to end; the material falling thru these openings will be collected and fed back to the furnace.

When all the strands of wire are in place and connected, therefore, in series, the current will pass zig-zag-wise in and out of the furnace and a wire resistance of considerable length will thus be afforded. The entire system forms a resistance grid on a very large scale.

As the heating-up is necessarily rapid and as no useful purpose can be served by maintaining the full temperature once it has permeated the wire, it is probable that a very short furnace will suffice, in which case it would be necessary to alter the design of the plant in order to afford the wire a sufficient length of time to cool in a non-conducting medium and out of reach of the air. As it leaves the furnace the wire might be led to the take-up reels thru tubes, in order to prevent chilling.

If, for any reason, a wire breaks or pulls out, the simplest thing to do is to cut it out entirely and connect up the sheaves between which it ran by a copper conductor or a standard resistance on the outside, until such time as the furnace is closed down for cleaning, inspecting or repairing.

The details of the operation may be described as follows: The

wire is quickly brought to a bright cherry-red, at which temperature all grease is volatilized, scale or rust is reduced by the carbon and occluded gases are driven out; at the same time, the zinc oxide in immediate contact is reduced and, as vapor, soaks into the "pores" of the metal, which are well opened by the heating, and condenses on the surface as the heat drops. As the wire returns thru the upper part of the furnace it cools down slowly from a temperature considerably above its recalescence point, so that it is annealed. With low-carbon steels an excess temperature of annealing of two or three hundred degrees is not likely to seriously affect the quality of the wire. The coating is of the color of freshly electro-deposited silver. The quality of the product is mostly due to the alloy of zinc and iron; as I have pointed out elsewhere, iron which has absorbed an almost infinitesimal amount of zinc by being exposed to zinc vapor in a reducing atmosphere, is most difficult to corrode and resists a solution of hydrochloric acid in a most remarkable manner.

The oxide and finely-divided carbon form a non-conducting mass, and the heat emanating from the wires will accumulate in the furnace until a constant temperature, not far below the reduction point of the oxide, will be reached; this will greatly reduce the amount of current required to operate the process. After the operation is under way, the resistance of the hot wires will be much greater than that of the cold wires.

Most of the time consumed in "sherardizing" is spent in forcing the heat from the outside thru a non-conducting mass of zinc dust; in the meantime, most of the heat-units go up the flue. The thermal efficiency of electro-cementizing is better and will go far towards balancing the excessive cost of electric heat.

It appears, therefore, that the new process would combine in itself the annealing furnace, the cleaning process and the zinc bath and do away entirely with the fluxing and wiping. While the cost of the heating will surely exceed that of the hot process, at least until the furnace has reached its constant temperature, the costs of installation, maintenance and depreciation, taken in one with the other, will probably be lower than for the hot plant, the cost of the zinc will be much less, standing at about 3½ cents on a 5 cent spelter market with less waste and the labor cost

should be little if at all in excess of what it is at present; the wire will run more slowly but there will be a compensating increase in the number of strands.

A serious objection has been offered with regard to the temperature of the wire. The estimated maximum temperature of 900° C. is high, and there is undoubtedly danger of the wire breaking inside of the furnace or, at least, being elongated; most galvanized wires are of small gauge, No. 12, for instance, being a very important size. The driving of the contact sheaves is calculated to relieve the tension created by the take-up reels and breakage from sag need hardly be feared and could easily be prevented. However, the criticism is a very legitimate one; trial on a commercial scale will reveal the problem, if it exists, and it can then be attacked and, I believe, successfully solved. The mechanical and electrical elements of the process are by no means complicated, and the most valuable criticisms and hints will come from those who know, from their experience with zinc reduction furnaces, what is likely to take place inside the coating chamber.

Oxides of other metals besides zinc may be used. Zinc-dust, with which I carried out my first test, deposits zinc at a much lower temperature, and might be useful for wire which must not be annealed, but its use is open to many serious objections. It is costly and by ageing it becomes in part converted into the carbonate of zinc, which requires a higher temperature of reduction at which, unfortunately, carbon dioxide begins to act and, unless special precautions be taken to maintain an atmosphere rich in carbon monoxide, the result is a gray coating of mixt zinc and zinc carbonate which suffers further decomposition, until, after a few months, it crumbles when the articles are hammered or bent. All dark-colored "sherardizing" is more or less in this condition, and the sulfate of copper test, to which inspectors cling as to a fetish, is worthless as a test of quality, its limited value being in connection with coatings of zinc and not of zinc carbonate. Again, the use of zinc-dust requires an air-tight furnace, which, in itself, would suffice to make the process uncommercial.

Zinc oxide, on the other hand, having a zinc content of 80 per cent., is an intermediate product in the manufacture of spelter, does not require an air-tight furnace, is not injurious to the work-

ers, like zinc-dust, and does not deteriorate, nor, on account of absorption of hydrogen from moisture, call for restrictions in respect to its storage, by fire-insurance companies. Most important of all, its reduction yields a coating of pure distilled zinc which is thoroly alloyed to the metal underneath.

Paris, France,
24th September, 1909.

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LAWS OF ELECTRODE LOSSES IN ELECTRIC FURNACES.

By Carl Hering.

At the last annual meeting of this Society the writer showed how it is possible to obtain in practice the combined effects of perfect heat insulation and good electrical conductivity, two antagonistic qualities which it was heretofore generally supposed could not both exist in the same material. As the electrodes of furnaces should be good electrical conductors, they also naturally have the very objectionable quality of being good heat conductors, as the one property generally accompanies the other. Hence, a way in which furnace electrodes of good electrical conductors may be made to also act as good heat insulators is of interest and importance to furnace designers. Based on this method, the writer has developed the laws according to which electrodes should be designed in order to reduce the loss of power and the amount of electrode material to the least possible. The purpose of the present paper is to enunciate these laws, illustrate their application and show their derivation. The results are in some respects quite different from what has generally been supposed, hence, economies, which sometimes may be quite great, will become possible in many if not in most cases.

The method above referred to is as follows: Let *e* Fig. 1 be the electrode. If now it be so proportioned that the electric or C^2R energy heats the furnace end to a slightly higher temperature than that of the furnace, there will be a flow of heat from that end *into* the furnace. Let the section *a* very near the hot end be that at which the temperature is greatest; then the heat gradients *cb* and *cd* will evidently *fall* in both directions; this means that some heat flows from that hot section into the furnace; and as heat cannot flow in two opposite directions at the same time, it follows that no furnace heat will flow out of

the furnace through such an electrode, which therefore becomes in effect a perfect heat insulator, more perfect even than the walls of the furnace, while at the same time it remains a good electrical conductor. In practice it is not necessary to make this temperature higher than that of the furnace, as it need merely not be lower, and the section *a* is best made to be the extreme end of the electrode. Hence the results will be accomplished if the C^2R loss heats the end of the electrode to the furnace temperature. It will be shown below that these are the conditions for obtaining the minimum loss.

There are two kinds of losses which must be considered in electrodes: First, the loss due to the conduction of heat by the

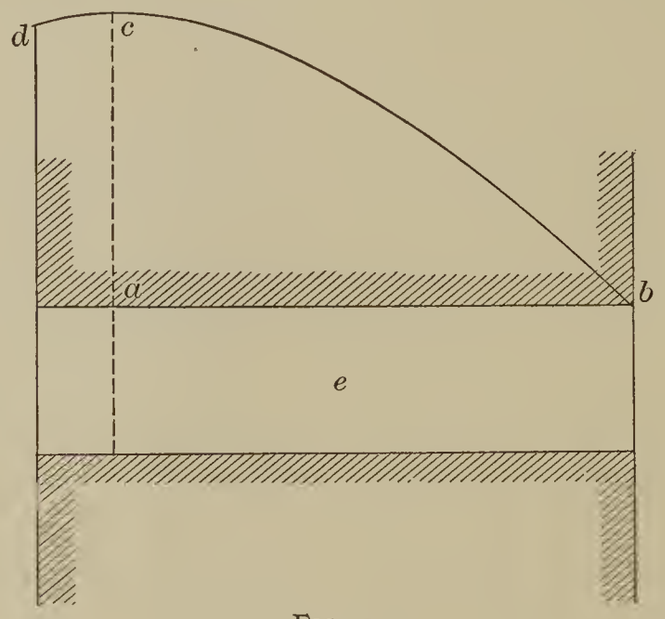


FIG. 1.

electrode from the material or gases in the furnace to the outside, which may be termed the heat conduction loss, or simply the thermic loss; and, second, the loss due to the electrical resistance of the electrode, which may be termed the electrical loss, or the C^2R loss. Both losses originate electrically, and are therefore of equal commercial value in cents per k. w. hour, and both leave as heat and are therefore thermic; but the above distinction is useful for differentiating between them. For a given length, if the cross-section increases, the heat loss increases, while the electrical loss diminishes; while for a given cross-section the reverse is the case when the length is increased. Moreover, the thermal and the electrical conductivities of the particular

material enter into the determination of these losses; and the total loss may be partly thermal and partly electrical, or entirely electrical or thermal, or even only a part of the C^2R energy. As these physical properties of the electrode material are an important factor, a comparison between two materials, like graphite and carbon, is evidently not a fair one unless each of the electrodes has its best proportions. The problem of finding the best conditions is therefore not a simple one.

The writer has therefore made a complete analysis of the subject of how an electrode of any material should be proportioned so as to obtain the least combined thermic and electric losses, with the least cost of electrodes. It will be found that such an analysis changes some of our preconceived ideas and leads to some unexpected results which may at first sight appear unacceptable because they are so different from our usual ideas about electrodes, but as they are based on the undisputed laws of heat conduction, their correctness must be accepted. The results of this analysis are published in the Electrochemical and Metallurgical Industry for October, 1909; in the present paper they are rearranged and amplified.

The writer has thus found the following relations to exist, which, as they are absolute, under the stated premises, may be termed the laws for electrode losses, for those assumed conditions.

Laws of Electrode Losses.

a. The combined loss through the cold end of an electrode is equivalent to the sum of the loss by heat conduction alone (when there is no current) and half the C^2R loss.

b. This combined loss will be least when the loss by heat conduction alone is made equal to half the C^2R loss; the total loss will then be equal to the C^2R loss, and no heat will be conducted from the interior of the furnace.

c. This minimum loss is dependent only on the material, current and temperature, but not on the absolute dimensions; it merely fixes the relation of the cross-section to the length, but leaves a choice of either; hence

d. For economy of electrode material the length should be made as short as practical considerations permit.

e. For each material there is a definite minimum loss of electrode voltage which depends only on the temperature and is independent of the dimensions or the normal current for which the furnace is designed; hence

f. The best possible electrode efficiency for any material may be determined from the total voltage of the furnace and this minimum voltage due to the material and the temperature, and is independent of the dimensions.

g. The temperatures indicated by the heat gradient of the combined flow are equal to the sums of those of the individual flows.

These laws are based on the following fundamental premises, any departures from which must be treated as correction factors. That the electrodes have a uniform cross-section throughout their whole length. That no heat leaves the electrode to the

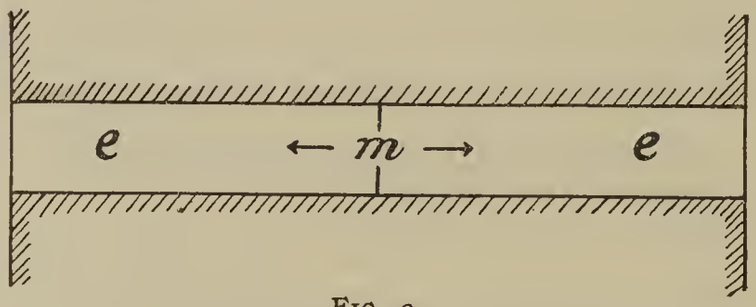


FIG. 2.

furnace walls or enters it from them; this is approximately true in most cases because the heat gradients are approximately the same in both, and there is contact resistance between them which may be quite great. And that the temperature variation of both conductivities may be neglected by using their average values instead. Both the two latter conditions may not be accurately true, but the differences are probably small in comparison with the best possible degree of accuracy with which the most desirable proportions of furnaces can be predetermined or realized in practice. Hence great accuracy is not essential. The matter of importance for us to know is in what direction to go to reduce the losses, and approximately what the minimum possible loss is, so that we can approach it as closely as practicable and do not waste efforts in trying to realize something which is impossible.

The conditions of minimum loss for a given normal current are illustrated in the following simple cases: Let *ee* Fig. 2 be

an electrode surrounded by a perfect heat insulator except at its ends, which are kept cool, say, with a water jacket. Let a relatively large current be passed through it. This will heat it to a high temperature at the middle *m*. When the stable state is reached, all the C^2R heat will flow out at the two ends, and none will flow across the middle section *m*.

If now this embedded electrode be imagined to be cut into two parts at *m*, and if these two parts are made the two end walls of a furnace with these conductors as its electrodes, as in Fig. 3, the temperature of the interior of the furnace being the same as that at *m* was in Fig. 2, it is evident that the heat flow will not be altered while the same normal current is flowing, and that therefore there will be no loss of heat from the interior of the furnace through the electrodes.

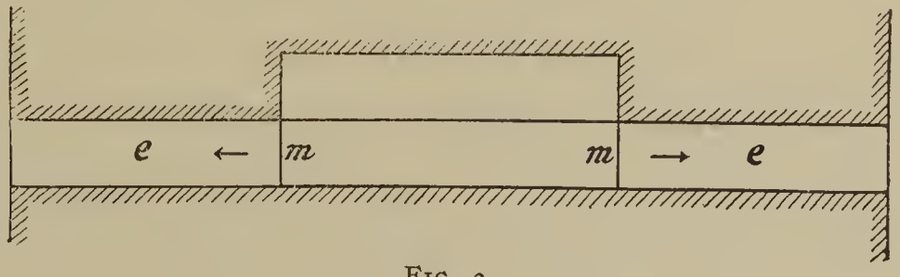


FIG. 3.

That this is the condition of minimum electrode loss will be shown later. It is rational to suppose that it would be so, because the energy of all the furnace heat which is lost through the electrodes must first be introduced into the furnace electrically, hence why should it be introduced at all if it flows right out again; it is somewhat like pumping an excess of water into a tank which is overflowing. Moreover, the electrode will normally have very nearly this temperature due to conducted heat, and all that the current does is to heat it a trifle hotter. It should also be remembered that this does not necessarily mean increasing the C^2R loss to what at first thought appears abnormal, but it merely means reducing the cross-section so that with a given C^2R loss this temperature will be reached. If with a given C^2R loss the section is made greater than this, the total loss will evidently be increased by the amount of the flow of furnace heat; while if the section is made less, the electrode will

have a higher temperature somewhere inside the furnace walls, as in Fig. 1, and the distance from there to the cold end is therefore less in addition to the temperature drop being greater, both of which increase the loss. Hence even without a mathematical proof it seems rational that this should be the condition of minimum loss.

The difficulty in calculating the results lies in the fact that no one seems to have determined in what way these two heat flows are to be added together to give the total. It is evidently incorrect to calculate each separately and add them, because two quantities of heat flowing together in the same conductor change the temperature conditions under which each was calculated; and, moreover, as was shown in Figs. 1, 2 and 3, it is possible to have one of them act as a check valve, so to speak, to stop the other from flowing.

Hence it is essential to first determine from the laws of heat the law by which these two heats are added. It will be noticed that they are essentially different from each other in that the furnace heat remains the same in quantity as it enters, flows through the electrode and departs, like a stream of water through a pipe. But the C^2R heat is generated in the inside of the electrode; only a little of it is flowing near the hot end, only half of it flows through the middle, and all of it flows out at the cold end, some of it having traveled only a short distance. It is like a porous pipe plugged up at one end and having water forced into it through its sides along its whole length, all of which flows out at the open end.

The writer has therefore determined the following general equation for the combined flow, the proof of which will be given later:

Let X be the total energy expressed in watts, which flows out at the cold end of a conductor; C the current in amperes; T the difference in temperature in centigrade degrees between the hot and the cold ends; S the cross-section in square inches; L the length in inches; k the average heat conductivity (or specific heat conductance) in gram calories per second per degree Centigrade for 1 inch length and 1 square inch section for the given range of temperatures; r the average electrical resistivity (or

specific resistance) in ohms for 1 inch length and 1 square inch section for the given range of temperature; and let 1 calorie per second equal 4.186 watts.

Then

$$X = 4.186\ kT \frac{S}{L} + \frac{C^2 r}{2} \cdot \frac{L}{S} \qquad (1)$$

It will at once be seen from this that the first term represents the heat (in watts) that would flow by heat conduction alone, that is, when no current is flowing; the constant 4.186 is merely the figure which reduces calories to watts. It will also be seen that the second term represents the C^2R loss and is equal to half of it. Hence this general law may be stated in words as follows: *The total flow of energy out of the cold end is equivalent to the sum of that which would flow by heat conduction alone, and half of that due to the electrical resistance.*

Conditions of Minimum Loss.

For a given normal current at which the furnace is to be operated, it will be noticed from this formula (1) that when, by proper proportioning, the heat conduction loss while no current is flowing (the first term), is made equal to half the C^2R loss (the second term), then the two terms are equal, and their sum is therefore equal to the total C^2R loss. It will be shown below that this is the condition of minimum loss described above.

For a given length the first term (heat conduction) increases as the section increases and in direct proportion, hence in Fig. 4 this quantity is an increasing one, represented by an inclined straight line *oh*. The second term, however (half of the electrode loss), will decrease as the section increases, and in inverse proportion, hence this quantity is a decreasing one, represented by the hyperbola *ee*.

The sum of two such quantities always has a minimum value, as is shown by the curve *mm*, which represents the sum of the two ordinates, and is therefore the curve of the values of X (in watts) in formula (1). It will be seen that this minimum point is reached when the two quantities are equal to each other, that

is, at the point where the two lines intersect; it is therefore at this point that the conduction loss is equal to half of the C^2R loss, which is the condition stated above.

If the same sets of curves be drawn for several other lengths of electrodes, as shown in dotted lines in Fig. 4, it will be noticed that the total loss at the corresponding minimum point always has the same value, namely, the height of the upper curve measured at its minimum point. This means that the minimum loss is determined only by the material, the temperature and the current, and cannot be further diminished by changing the dimensions.

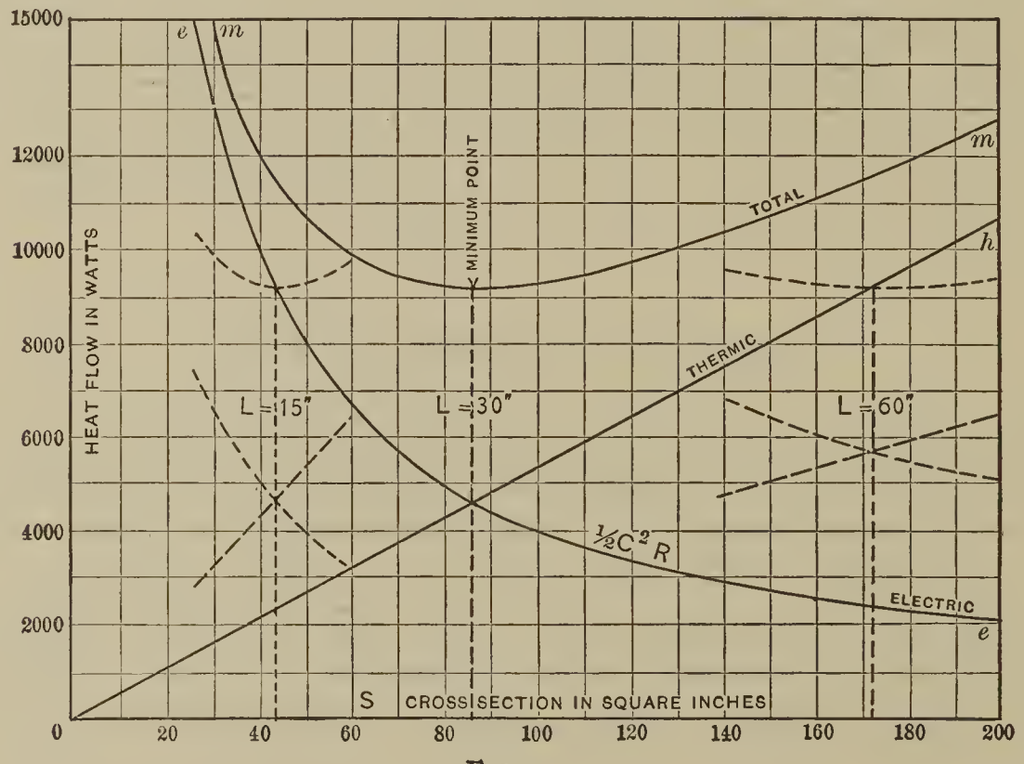


FIG. 4.

It is therefore a fixed quantity, determined by the given conditions of material, temperature and current, and no possible change of dimensions can reduce it. To reach this minimum requires a certain proportion between the length and the cross-section, as will be discussed later, hence no further change of dimensions can possibly reduce it.

Let mX be this minimum total loss in watts. Making the two terms in formula (1) equal, solving for the ratio of S to L and substituting in (1) gives

$$mX = 2.894\ C\ \sqrt{krT} \qquad (2)$$

from which the minimum loss in watts can readily be calculated for any given case. The constant 2.894 is a purely mathematical one and includes no physical properties.

It is of considerable interest to note that this formula shows that this loss is independent of the dimensions and is fixed by the current, temperature and material (k and r). It is also of interest to note that it increases in direct proportion with the current and not with its square; also that it is independent of the amount of energy in the furnace, being a function merely of the current, which means that for a given input in kilowatts in the furnace, the minimum total electrode loss becomes smaller if the current is decreased and the voltage increased. For a given normal current, material and temperature, the conditions are fixed, and the electrodes may be determined; the minimum loss then, of course, applies to this normal current and to none other; for if with the electrodes thus determined the current falls below this normal, the loss will, of course, be less, but so will also be the energy in the furnace. What is said above, of course, applies only to the normal current specified for operating that furnace.

The formula also shows that the minimum electrode loss increases only as the square root of the temperature, and not as the temperature itself, as might at first be supposed. This means that the temperature may be increased considerably without increasing the loss greatly.

The same is true of the heat conductivity k and of the electrical resistivity r, either of which must be decreased considerably in order to better the loss appreciably. Hence the choice of material is not as important a factor to reduce the loss as might at first be supposed, and such choice may therefore often be determined by other consideration such, for instance, as size, convenience in handling, size of water jackets, terminals, etc., all of which will be shown to be far more favorable for graphite, for instance, than for carbon, although the minimum possible loss of energy seems to be slightly greater than for the latter. It will also be found that this minimum loss is in most cases quite small, much smaller perhaps than is generally supposed, hence a further reduction by reducing any of the determining factors in formula (2) should be carefully considered with respect to any possible

attending disadvantages, chiefly as to size, as it may not be worth making. To reach this minimum loss with carbon, for instance, requires the electrodes to be far larger in section than for graphite (for a given length), and if not made of this much greater size the loss will not be the minimum and hence may then exceed that of graphite. The differences between the minimum losses in these two materials (provided the best proportions can be realized in practice, which may not always be the case with carbon) does not seem to be great, yet the difference in the size is very great, as will be shown below.

Another feature should be considered: In high temperature furnaces carbon may graphitize partly, and if it was correctly proportioned at first, it will evidently not be so later on, hence the loss will then become greater again. Graphite is stable and does not change, hence if correctly proportioned at the start, it remains so.

The resistivity r may be replaced by the reciprocal of the electrical conductivity K, and for comparing the qualities of materials as such, the current and temperature may be made unity; the result will then represent the loss in watts per ampere for one degree of temperature, and will therefore be characteristic of the material itself. Making these substitutions gives

$$mX' = 2.894 \sqrt{\frac{k}{K}} \qquad (3)$$

K is in reciprocal ohm (mho), cubic inch units.

This shows the interesting fact that the minimum loss depends on the *ratio* of the two conductivities—both may be large or both small and yet give the same result as far as the loss is concerned. The smaller this ratio, the better the material, as far as the minimum loss is concerned. Moreover the loss varies with the square root of this ratio, which must therefore be considerably diminished in order to reduce the loss materially.

In order to improve materials for electrodes when this minimum loss is the criterion, the electrical conductivity should be increased as much as possible and the heat conductivity diminished as much as possible; but it is their ratio that is the deciding factor, hence the heat conductivity may even be increased and yet the

ratio may be less, if the electrical conductivity has increased still more in proportion.

There is still another interesting feature about this minimum loss. The loss represented by the formula (2) is also equal to Ce in which e is the drop of voltage in one electrode. Making this substitution gives

$$e = 2.894 \sqrt{krT} \qquad (4)$$

which is seen to be independent of the current, and which therefore means that for every material (k and r) there is a characteristic minimum drop of potential in the electrodes for one degree of difference of temperature, below which voltage it is not possible to go without again increasing the total loss; and this is independent of the capacity, the dimensions, and even the amount of the normal current, the total drop being dependent only on the temperature. It might be termed the electrode voltage of that material, as it is a characteristic property of it as an electrode material.

This may be stated in another way. If R is the resistance of one electrode in ohms, then $e = CR$ which combined with (4) gives

$$R = \frac{2.894}{C} \sqrt{krT}$$

which is the minimum resistance (in ohms) which an electrode of any given material (k and r) should have; if it is made either less or greater than this, the total loss is again increased. As this value of the resistance depends also on the current, it is not as much a characteristic of the material as the minimum electrode voltage just described is. It might be said to be the minimum resistance per ampere, but this is only another name for the drop in voltage. For any given furnace the current is generally fixed by specification, and in such cases the resistance also becomes a fixed quantity, either for one degree temperature or for any given temperature.

If by the term electrode efficiency is meant the voltage between the two inside or hot ends of the electrodes, divided by the voltage between the two outside ends, its highest possible value is determined by the total voltage of the furnace, the temperature and

the material, and is independent of anything else. Hence for any given electrode material and temperature drop, the furnace voltage necessary to give any desired electrode efficiency may readily be calculated.

This minimum electrode loss is not merely a theoretical ideal which it is impractical to reach, but it is an eminently practical result which could probably be readily reached in most furnaces, with graphite, at least, if not with carbon also, when the data for its proper determination, namely, the two conductivities, exist. Attention should, however, be called to the flatness of this minimum curve *mm* near its minimum point, which means that great accuracy in the proportioning is not necessary, as slight variations in the proportions in either direction will make only a small difference in the total loss. For instance, in the case shown in Fig. 4, a change of 10 to 20 per cent. in the cross-section near the minimum point will increase the loss only about 1 to 3 per cent.

Proportions and Economy of the Electrodes.

Although the absolute dimensions of the electrodes are not fixed by the conditions for obtaining the minimum loss, yet the relation of the section to the length is. This is best shown analytically. If the two terms in formula (1) are made equal, which is the condition of minimum loss, the ratio of S to L is found to be a constant, determined by the material (k and r), the temperature and the current. That is, when

$$4.186\ kT\ \frac{S}{L} = \frac{C^2 r}{2} \cdot \frac{L}{S}$$

then

$$\frac{S}{L} = 0.3456\ C\ \sqrt{\frac{r}{kT}} \qquad (5)$$

This means that to get the minimum loss under given conditions of material, current and temperature, the cross-section must merely bear a certain relation to the length as given by this formula. The interesting conclusion from this is that any number of different electrodes will give the same result, as illustrated in Fig. 5, in which all have the same ratio of section to length, as required by formula (5). The cross-section in all

these is 10 times the length in inch units. It thus leaves one a choice of either the length or the section, but not of both, and this choice does not concern the loss of power—it merely affects the economy of electrode material. Practical considerations usually determine the length, and it is therefore generally the cross-section which is to be determined from formula (5).

Notice should be taken of the fact that the numerical value of this ratio will be different for different units, as, for instance, for inches and for centimeters; the actual sizes will, however, of course, be the same.

The rule concerning the economy of electrode material is therefore simply: *Make the electrodes as short as practical con-*

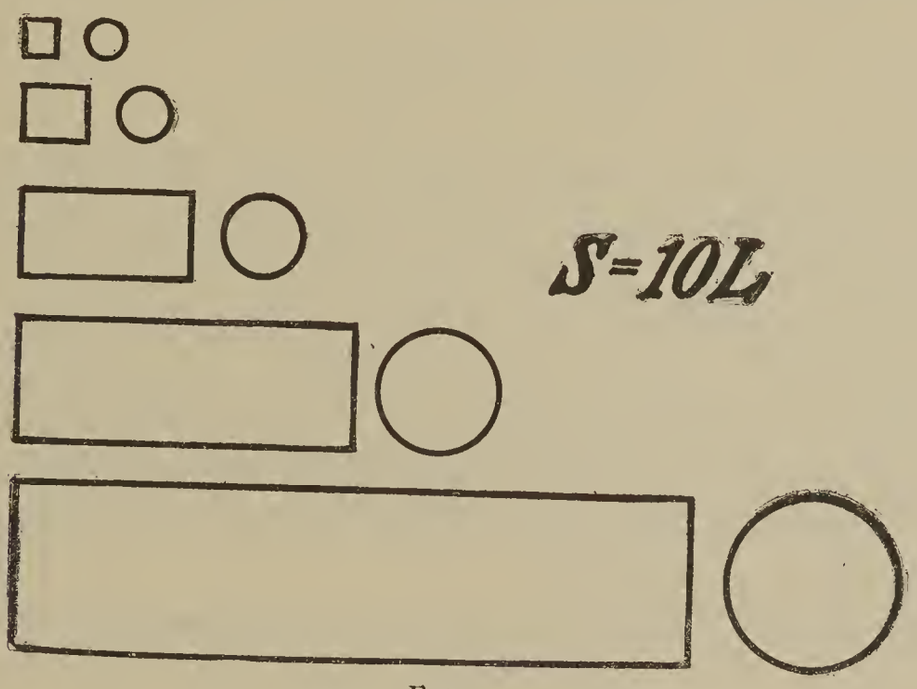


FIG. 5.

siderations will permit. As the cross-section also diminishes with the length, the volume or approximately the cost in dollars diminishes as the square of the length. Hence the great importance of making the electrodes as short as possible, when the economy of the electrode material is of importance; this choice of length will not affect the power loss and can therefore be decided on its own merits and without regard to the power efficiency.

For a given length the formula may be written in the form

$$S = 0.3456\, CL \sqrt{\frac{r}{kT}} \qquad (6)$$

which means that for a given length, temperature and material, the section increases in direct proportion with the current; hence the importance of producing the required heat in the furnace by as high a voltage as possible, so as to diminish the normal current. This will not only make the electrodes smaller in the direct proportion as the current is diminished, but, as was shown above (2), it also diminishes the lowest possible loss of power in the electrodes; hence it is a double gain.

It will be noticed from (6) that the section diminishes as the square root of the temperature, that is, for higher temperatures it becomes smaller.

Letting the current density in amperes per square inch be represented by Cd, then formula (5) reduces to

$$Cd = \frac{2.894}{L}\sqrt{\frac{kT}{r}} \qquad (7)$$

which shows that the proper current density to obtain the best possible proportions varies inversely with the length, and therefore directly as the section; hence it is quite incorrect to proportion electrodes on the basis of a fixed current density, as is often done. For any assumed length the resulting current density can readily be calculated from this formula so as to compare it with the maximum which the material will stand; if water cooled at one end, it will stand much more, hence water cooling results in an economy of material.

Making C and T equal to unity in formula (5) gives

$$\frac{S}{L} = 0.3456\sqrt{\frac{r}{k}} \qquad (8)$$

which shows that per ampere and for one degree temperature there is a definite best proportion of section to length for each material which is a characteristic of that material and below which it is not possible to go in economizing without increasing the loss. Hence this figure affords a good means of comparison of different electrode materials when the economy of the material is the criterion. It shows that in this respect graphite is far superior to carbon, as electrodes of the former are very much smaller, as will be shown later.

Substituting in (8) the electrical conductivity K for the

resistivity r, as was done in formula (3) for the convenience in making comparisons, this formula (8) reduces to

$$\frac{S}{L} = 0.3456 \sqrt{\frac{1}{Kk}} \qquad (9)$$

This shows that, as far as economy of material is concerned, that material is best in which the *product* of the two conductivities is greatest. Hence to improve a material in this respect either or both should be increased. But the improvement in economy is only in proportion to the square root and hence is not as great in percentage as the improvement in either of the conductivities.

Comparing this formula (9) with the corresponding one (3) for the minimum loss, it will be noticed that the requirements for economy in material (9) and for economy of power (3) are different, and in some respects antagonistic, hence the choice of the material depends somewhat on the relative importance of these two economies. With graphite and carbon, for instance, according to the best though crude figures available for the two conductivities, the electrodes become much smaller for graphite, but the minimum loss is slightly less for carbon, provided both have their best proportions and, of course, the same length. Hence the question arises: Is it worth while to use the far larger carbons than the much smaller graphites in order to diminish an already low power loss slightly? Numerical examples will be given below.

In this comparison of (3) and (9) it will be seen that both economies are improved by an increase of the electrical conductivity alone, hence this is always desirable. An increase in the heat conductivity alone improves the economy of material but diminishes the economy of power; a decrease in the heat conductivity alone improves the economy of power but increases their size. The final criterion depends on their quotient and product, as was described.

In formulas (5), (8) and (9) the relation is stated in the form of section divided by length; this was done because practical considerations usually fix the length, and it is therefore the cross-section which remains to be calculated. By inverting these formulas so as to give the ratio of the length to the section they become more similar to (2), (3) and (4), with which they can

then be better compared for making further deductions. Thus when the chief formula (5) is innverted it becomes

$$\frac{L}{S} = 2.894 \frac{1}{C} \sqrt{\frac{kT}{r}}$$

in which the numerical coefficient is the same as in (2), (3) and (4).

Rules for Designing Electrodes.

From the above the rules for the guidance of the designer may be briefly summarized as follows:

The temperature and the kilowatts are always given in the problem. The two conductivities or resistivities of the proposed materials must, of course, be available. From the kilowatts make the current as small and the voltage as high as practicable. From formula (5) calculate the proportion of the section to the length. The length is generally fixed by the width of furnace walls or other considerations; the only rule is to make it as short as practicable; this length then determines the section. The current density (7) is not a factor in calculating the proportions of electrodes except insofar that it should not exceed practical limits.

The electrode loss will then be the minimum possible, and is determined from formula (2) in watts; this is for one electrode and must therefore be multiplied by 2 to get the total. The electrode efficiencies are then readily determined.

These calculations should be repeated for each of the different materials available and the various advantages and disadvantages due to their different sizes, losses, terminals, etc., should be carefully weighed.

Never compare two electrodes of the same size but of different materials having different conductivities; such a comparison is unjust to either one or the other or both, as also to the designer, because it gives him an unwarranted confidence. They can be properly compared only when both have their best proportions.

Numerical Examples.

To give numerical examples for illustrating these laws of electrode losses and their best proportioning requires that the two

conductivities be known for those ranges of temperature. These constants unfortunately do not all exist in satisfactory form, but now that the laws of the electrode losses have been definitely established, these conductivities will no doubt be determined. A simple method for determining them is given below. For the present, therefore, we must content ourselves with the best data that are available, but it must not be forgotten that the comparisons based on them are no more reliable than the original data, and that the present results are therefore only approximate. The numerical examples are given here chiefly as illustrations and rough approximations, and not as accurate and reliable figures. But they are, of course, far better than those determined by the former methods based on current densities, the addition of the two losses, and on a comparison of electrodes of the same size but of different materials.

The electrical resistivity of graphite is probably quite close to 0.000333 ohm for 1 inch cube, taken lengthwise. But this is at normal temperatures; it falls quite appreciable at higher temperatures,[1] reaching as low as about 64 per cent. A fair average for a range of temperature from 100° C. (water jacket) to 1700° C. (approximately that of iron furnaces) is therefore about 0.000266.

The absolute heat conductivity of graphite has apparently not been determined, notably not at the higher temperatures. According to FitzGerald it is 18 times that of carbon, while according to Hansen it is about 8 times carbon. Both are only relative and not in absolute units. The former figure seems to bring it up higher even than many metals and is therefore probably much too high; the second was determined under furnace conditions and is therefore probably more nearly correct. It will be so assumed here.

The heat conductivity of carbon is also not known. The only figure obtainable is 0.0103 in absolute units for retort carbon (about 0.026 in cubic inch units), which seems rather low for electrode carbons, which are certainly more like the metals than the vitrious retort carbons are. According to Hansen it decreases at higher temperatures. It is therefore probably not far wrong

[1] According to figures obtained by the writer through the kindness of Mr. C. A. Hansen.

to assume it to be 0.03 in cubic inch units. Graphite on the basis of 1 to 8 would therefore be 0.24.

The electrical resistivity of carbon seems to average about 0.00150 in ohm cubic inch units for the smaller sizes, but according to Hansen it increases to 0.00337 for 6 inches by 6 inches, and to 0.0055 and even 0.0100 for 18 inches by 18 inches. As carbon electrodes must be very much larger than graphite, a comparison between the two becomes fairer if some account is taken of this higher resistance for the larger sizes; it may therefore be assumed to be 0.0025, and, as with graphite, that it diminishes with the temperature, making the average between 100° and 1700° C., about 80 per cent. of this, which is 0.002.

Hence the constants to be used here are, in cubic inch units, for a range of 100° to 1700° C.: Graphite, $r = 0.000266$, $k = 0.24$; carbon, $r = 0.002$, $k = 0.03$.

From formula (2) it will be seen that the minimum loss, so far as the material is concerned, is proportional to the square root of $k \times r$, which for graphite is 0.0080 and for carbon 0.0078; hence the difference in the losses will be only about 3 per cent. in favor of the carbon.

Similarly from formula (5) the cross-section for any given length, as far as the material is concerned, is proportional to the square root of r/k, which for graphite is 0.0333 and for carbon 0.258; hence for any specified furnace carbon electrodes would have to have a cross-section $0.258/0.0333 = 7.75$, or nearly 8 times that of graphite for the same furnace. The question therefore arises: Is this very small gain in power worth the disadvantages of the enormously larger electrodes with their larger terminals, water jackets, etc.

Let the capacity of a furnace be 500 kw. and let it be decided to run it at 50 volts and 10,000 amperes, the temperature to be 1700° C. inside and 100° C. at the outside ends of the electrodes. From formula (2) the minimum loss in each electrode will therefore be 9.15 kw. for graphite, hence the loss in the two electrodes together due to conduction and resistance is only 3.66 per cent. For carbon is will be 8.96 kw. or 3.58 per cent. It is to be noted how relatively small these actual losses are and what a small difference there is between graphite and carbon

when they are properly proportioned. This difference is about 3 per cent of 3.5 per cent, or about one-tenth of 1 per cent. of the total power. The curves of Fig. 4 are for the graphites for this furnace.

From formula (6) the cross-section for graphite in square inches will be 2.87 times the length in inches, and for carbon 22.3 times. Let it be determined from practical considerations that the length should be 30 inches, and let the electrodes be round in section, then the diameter of the graphite will be 10.5 inches,

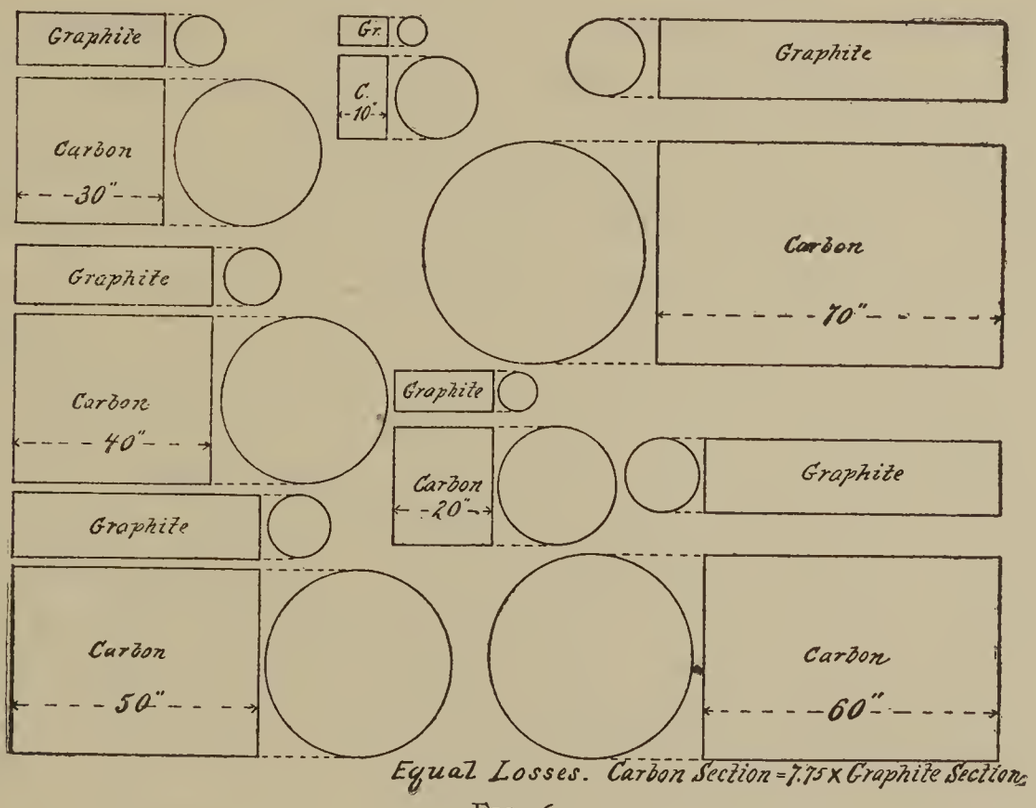


FIG. 6.

and that of the carbon 29.2 inches. As stated above, the carbons will have nearly 8 times the cross-section of the graphites for the same length.

It will be seen that the carbon electrode will be about as large in diameter as it is long and is therefore entirely impracticable. For a length of 40 inches the diameters are 12.1 inches and 33.7 inches respectively; for 50 inches, 13.5 inches and 37.7 inches; for 60 inches, 14.8 inches and 41.3 inches, and for 70 inches, 16 inches and 44.6 inches. In order to give a better idea of these proportions they have been drawn to scale in Fig. 6 in pairs of the same length. For all these electrodes of the same material

the loss is the same, that for the carbons being about 3 per cent. lower than that for the graphites.

It will be seen what an awkward and entirely impracticable proportion the carbons have for such a furnace. To lengthen any of them appreciably for the same section would increase the loss, and as the difference in loss is only 3 per cent., it would soon become greater than for graphite.

Hence the prevalent idea that the loss with graphite electrodes is always too great owing to their high heat conductivity is a fallacy and is based on an improper comparison. As a matter of fact, it is their good heat and electrical conductivities that enable them to be made small and of such proportions that it becomes possible to obtain the minimum loss for such a large current furnace which is it evidently impossible or at least impracticable to obtain with carbons, at least for this furnace, provided the assumed values of the conductivities are correct.

This illustration also shows quite strikingly the error committed when two electrodes of the same size but of different materials are compared with each other. Either one or the other, and probably both, must have been badly proportioned; it was no doubt generally the graphite which was much too large and therefore naturally chilled the furnace. One can readily convince himself that these are the best proportions by changing either the length or the section, when it will be found by calculation that in every case the total loss will be increased. Formula (1) shows how this total loss may be calculated.

The designer of this furnace, therefore, has his choice of all the shapes and sizes in this Fig. 6, or any others in which the same relation between the section and length exist. If, in addition to the awkward shape of the carbons, the extra cost of the larger terminals, water jackets, etc., are considered, there is little doubt that graphite would be preferred for this furnace, if the cost is not prohibitive. The choice between these different graphite electrodes is determined by the economy of electrode material, the size of the terminals, the size of the furnace walls, etc., which means that the one having the least practical length should be preferred. The quantity of electrode material is inversely as the square of the length. In estimating the first cost, the greater loss of energy, which is a continual loss, should

also be considered, if it is appreciable, for it is evident, from the proportions in Fig. 6, that it is not always practicable to reach the minimum loss, as, for instance, with the carbons in this case on account of the awkward proportions, hence the loss for that furnace will probably be greater with carbons than with graphites.

The current densities in amperes per square inch in these graphite electrodes of Fig. 6 are as follows: 30-inch, 116; 40-inch, 87; 50-inch, 70; 60-inch, 58; 70-inch, 50. Hence they are not at all excessive; on the contrary, even the highest is quite low, and perhaps even a shorter one would answer. The objection to making it much shorter is that it becomes too chunky, that is, short and fat, like the carbons. This is shown in the small pair of figures at the top, in which the length is only 10 inches; the current density in this short graphite electrode is 350. The proportions for a length of 20 inches have also been added, as it might be practicable to make the graphites as short as this.

In comparing the quantities of electrode material (that is, the cost), one should, of course, compare the short graphite one with the much longer carbons, because the latter cannot be handled unless made sufficiently long. If, for instance, the shortest practicable carbon is 40 inches long and graphite 20 inches, the quantity of material in the former will be nearly 32 times as great. The drop of voltage in these electrodes, which is the same in all those of the same material, is determined from formula (4), or more directly from the above minimum loss, which was found to be 3.66 per cent. for the graphite; hence $.0366 \times 50. = 1.83$ volts, or 0.92 per electrode; and for the carbon 3.58 per cent., or .90 volt per electrode.

Formula (4), of course, gives the same results, but to illustrate its application as giving the characteristic voltage drop of the material, substitute the constants for graphite and carbon, and make the temperature unity. This gives for graphite $e = 0.0231$ and for carbon $e = 0.0224$ as the characteristic minimum voltage drop for one degree for these materials. These are independent of the normal current for which the electrodes are designed; that is, they are the least voltage drop for any normal current, and are for one degree difference of temperature. Hence the minimum voltage drop can be determined for these

materials at once by simply multiplying these figures by the square root of the temperature. In the present case the temperature drop is 1600, hence multiply by 40, which gives .92 and .90 as the two drops in voltages per electrode, regardless of which of the sizes of the electrodes are chosen.

It might be asked, what will be the result if the cross sections and lengths are made the same for both materials? The answer is, if the carbon is in that case the one which is properly proportioned, then the one of graphite will be far too large in section and will therefore chill the furnace. If, on the other hand, the graphite is the properly proportioned one, then the carbon will become much hotter than the furnace at some place within the furnace walls, like in Fig. 1. This means that the carbons are unnecessarily strained and that the loss is greater than it would be with even a shorter carbon; hence it means a waste of both power and electrode material.

It may also be asked, what if the sections are the same and the lengths different, so that both are correctly proportioned for the minimum loss? The answer is that the graphite would then be very much larger than necessary, it being many times the length of the carbon, and it would be absurd to make it so, as a much shorter and smaller one would give just as good a result.

Lastly, it may be asked, what if they have the same length and the same loss, the graphite being the one which is proportioned correctly? There are then two different cross-sections of the carbon which fulfill these requirements. The smaller one means that it will get hotter than the furnace and therefore ought to be shorter, and the larger one is larger than required for the minimum loss, hence it would be absurd to use it.

Hence the only way the slightly lower minimum loss for carbon can be taken advantage of is to proportion the carbons quite correctly.

It might be added that the furnace above taken as an illustration is a large one, 500 kw. For very small furnaces the proportions of the carbons become more practicable, while the graphites on the other hand, become very small in cross-section, which may at times make them awkwardly slender. But in the smaller furnaces it is likely that lower voltages, and therefore relatively greater currents would be used, and as the section of an electrode

is a function of the current, these apparent disadvantages of graphite again become less. The only way to decide is to calculate them both for each particular case, and if possible to proportion the voltage and current so as to favor a lower loss and a smaller volume.

Proof of the Fundamental Equation.

The proof of the equation (1) is as follows: Let *ab* Fig. 7 be a conductor of heat and electricity of uniform section, which is surrounded by a perfect insulator of electricity and heat except at its ends. Let the one end *a* be kept at a temperature T by some source outside of it, and the other end *b* be kept at zero, hence heat will flow through it from *a* to *b*. In addition to this, let an electric current flow through it which sets free heat in its interior at a uniform rate in watts per inch. The

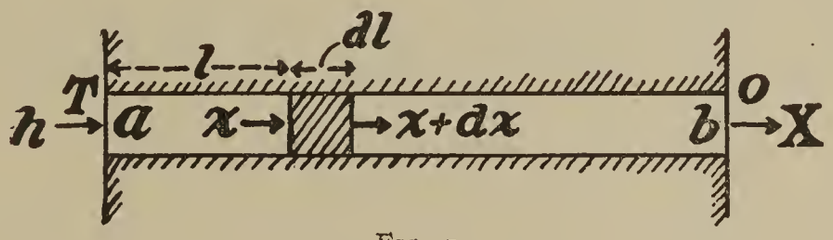


Fig. 7.

problem then is, how much heat will flow out of the cold end after the stable state has been reached.

Let:

X = total energy flow in watts passing out at the cold end.

x = total energy flow in watts passing through any section at a distance l from the hot end.

H = total heat flow in watts which would flow from the hot to the cold end when there is no electric current.

h = heat flow in watts which actually enters the hot end.

W = total heat flow in watts generated in the rod by the electrical current; that is, the total C^2R heat.

w = this C^2R heat per inch of length, = W/L.

T = temperature of hot end in centigrade degrees, when cold end is at zero, or the drop of temperature when it is not.

t = temperature at any given section at a distance l from the hot end, when the cold end is at zero.

L = total length in inches.

l = any given distance from the hot end, in inches.

S = cross-section in square inches.

k = mean heat conductivity in gram calories per second for one inch length and one square inch section, for the given range of temperature.

r = mean electrical resistivity in ohms for one inch length and one inch section, for the given range of temperature.

C = the current in amperes.

R = the total resistance in ohms.

j = 4.186, namely, the electrical equivalent of heat; that is: gram calories per second $\times$ j = watts.

Let dl be an infinitely short section at a distance l from the hot end, t be the temperature at the hot side of this section, and let the heat which flows into this section from the hot end be x. Then, as the heat flow follows some law, the heat which flows out of that section is x + dx. Hence the heat which has been added is the difference, namely, dx. But this heat, which has been added by the electrical current, is also equal to wdl. Hence

$$dx = wdl. \tag{10}$$

but

$$x = -jkS \frac{dt}{dl} \tag{11}$$

in which dt/dl is the heat gradient, which is negative because it increases as t diminishes. Differentiating this gives

$$dx = -jkS \frac{d^2t}{dl^2} dl.$$

which combined with (10) and reduced, gives

$$\frac{d^2t}{dl^2} = -\frac{W}{jkS}$$

Integrating this once, gives

$$\frac{dt}{dl} = a - \frac{wl}{jkS} \tag{12}$$

and a second time

$$t = b + al - \frac{wl^2}{2jkS} \tag{13}$$

Now when $l = L$ then $t = o$, and when $l = o$, then $t = T$. Substituting these values in this equation gives the value of the constants a and b, as follows.

$$b = T$$

$$a = \frac{wL}{2jkS} - \frac{T}{L}$$

Substituting this value of a in (12) gives the value of the differential co-efficient dt/dl in finite quantities; and this substituted in (11) gives the general equation for the flow of energy at any point l inches from the hot end:

$$x = \frac{jkTS}{L} - \frac{wL}{2} + wl \qquad (14)$$

It will be seen that the first term is the total conducted heat H in watts when there is no current, the second is ½ the total C^2R heat, that is, W/2, while the third term is the C^2R heat generated in the hotter part l. Hence the general equation for the heat flow through any given section is

$$x = H - \frac{W}{2} + wl. \qquad (15)$$

Or, in words, this means that at any point l inches from the hot end, the flow of energy is equal to the total heat which would be conducted by the whole rod from the hot end to the cold end in watts, when there is no current, less ½ the total C^2R energy per second in the whole rod, plus the C^2R energy per second generated in the part l.

Now when $l = L$, then $x = X$, because it is then equal to the total flow out of the cold end; hence, as $wl = W$,

$$X = H + \frac{W}{2} \qquad (16)$$

which is the fundamental equation (1) because the heat which would be conducted from the hot end is

$$H = jkT\,\frac{S}{L} \qquad (17)$$

and half the C^2R loss is

$$\frac{W}{2} = \frac{C^2r}{2} \cdot \frac{L}{S}. \qquad (18)$$

Hence this fundamental equation means that the total energy flowing out of the cold end is equal to the sum of the heat which would flow if there were no current, and half the C^2R heat.

This equation may also be deduced by ordinary algebraic methods. It has also been checked by slightly different methods by Dr. H. C. Richards and Dr. W. S. Franklin; it was also determined by a slightly different method, quite independently, though subsequently, by Dr. E. F. Roeber, including the deduction made below concerning the condition of minimum loss.

Other Analytical Deductions.

If in (15) l is made equal to zero, then $x = h$; that is, the heat which enters the hot end is

$$h = H - \frac{W}{2}$$

or, in other words, the furnace heat which enters is equal to the difference between these two heats. Hence in order to make $h = 0$, that is, to have this rod conduct no heat from the source at the hot end,

$$H = \frac{W}{2} \qquad (19)$$

or, in other words, the heat which would be conducted if there were no current, must be equal to half the C^2R heat. This is accomplished by proportioning the cross-section and length so that this equality results, as was shown above.

Returning to the heat flowing out of the cold end (16), it will be noticed that the product of these two terms is, from, (17) and (18)

$$\frac{C^2 jrkT}{2}$$

which does not contain the variables S and L, and is therefore a constant, for any given conditions of a problem. But if the product of two variables is a constant, their sum (16) is a minimum when they are equal, and this sum X, is then equal to twice either one of them. Hence for the minimum flow of heat out

of the cold end, that is, for the minimum total loss of energy in an electrode of a furnace,

$$H = \frac{W}{2}. \qquad (20)$$

As was shown above in (19), this equality also exists when no heat enters the hot end of the rod; or in furnaces, when no furnace heat leaves through the electrode. The total loss is equal to their sum (16) but as they are now equal, this sum is equal to either 2H or to W. Or, put into words: *the total loss is least when the heat which would be conducted if there were no*

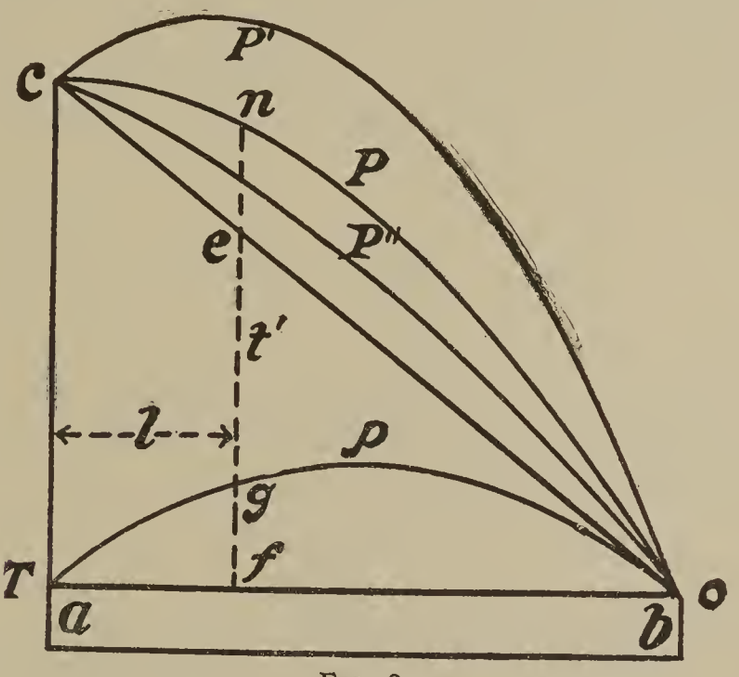


Fig. 8.

current, is equal to half the C^2R heat; there is then no loss of heat from the furnace; and the loss through the cold end is equal to the C^2R loss.

Returning now to the general equation for the temperature t (13), substituting in it the values of a and b given below it, and replacing wL by W gives

$$t = T - \frac{Tl}{L} + \frac{Wl}{2jkS} - \frac{wl^2}{2jkS} \qquad (21)$$

which is the general equation of the temperature, and therefore gives the heat gradient along the rod. It will be seen to be parabolic.

Making w equal to zero in this equation, that is, when no

electric current is flowing, the heat gradient is a straight line *ceb* Fig. 8 in which *ab* is the conductor and *a* the hot end.

Making T equal to zero in (21) gives

$$t = \frac{Wl}{2jkS} - \frac{wl^2}{2jkS} = \frac{(W-wl)\,l}{2jkS} \qquad (22)$$

which is the equation of the parabola *p*, Fig. 8; this therefore is the heat gradient for the case shown in Fig. 2 above, in which a rod is heated by a current while imbedded in a heat insulator, and is cooled only at the two ends.

It was shown above that the total loss of heat from the cold end is least when the conduction heat is equal to half the C^2R heat; that is, when the quantity (17) equals W/2. Making these two quantities equal, solving for T and substituting in (21) gives

$$t = \frac{WL}{2jkS} - \frac{wl^2}{2jkS} = \frac{w}{2jkS}(L^2 - l^2) \qquad (23)$$

which gives the parabola *P* Fig. 8. This therefore is the heat gradient for the case of minimum loss, which is the proper condition under which electrodes should be used.

Making l = 0 in (23), then t = T; hence

$$T = \frac{WL}{2jkS}. \qquad (24)$$

As this is the condition under which no heat flows into the hot end, this temperature is that due only to the C^2R heat W, hence this formula enables one to calculate the temperature to which the C^2R heat will raise a rod when no heat leaves it except at the one cold end. In practice this condition is realized in Fig. 2 when the electrode is considered to be half of the rod *ee*, from the middle *m* to either end.

Incidentally it might be added that this formula (24) enables one to determine the mean heat conductivity k of such conductors in a very simple way and without any heat measurements, by heating such a rod electrically as shown in Fig. 2, and measuring the current, the voltage and the difference of temperature between its middle point and its ends, when the stable state has been reached; the current multiplied by half the voltage between the ends gives the watts of heat W in one half; then by sub-

stituting these values in (24), remembering that L is now only half the length, the value of k is readily calculated. Practically perfect heat insulation may be obtained by surrounding the rod with a group of similar ones near it and parallel to it, forming a sort of squirrel cage around it, and heating them also to the same temperature, preferably with the same current; if all the cold ends are also kept at the same temperature the heat gradients will be the same in all the rods, and therefore no heat will flow from the test rod into the insulating material surrounding it, or the reverse. The current and voltage in this test also enables the mean specific electrical resistance to be calculated, thereby giving both the constants needed for proper calculation of electrodes. This method is further described in another paper by the writer, read at this meeting, entitled "A New Method of Measuring Mean Thermal and Electrical Conductives of Furnace Electrodes."

Solving equation (17) for T gives

$$T = \frac{HL}{jkS}. \qquad (25)$$

Comparing this value of T with that in (24) shows the interesting fact that when H = W the temperature at the hot end will be just twice as great when a given amount of heat enters at one end and flows out through the other, than when that same amount of heat is generated uniformly within the rod itself and all flows out of one end.

Returning to equation (21), its physical meaning may be interpreted by rewriting it

$$t = \left(T - \frac{Ti}{L}\right) + \frac{(W - wl)\ 1}{2jkS} \qquad (26)$$

it will then be seen that the first term in parenthesis is the temperature due to the heat entering the hot end and flowing through the rod, when no current flows; that is, the distance *ef*, Fig. 8, to the straight line *cb*. The second term is seen to be the same as (22), which is the temperature due to the C^2R heat alone, that is, when both ends are cold and there is no heat to be conducted except this C^2R heat; this is represented by the distance *gf*, Fig. 8. Hence the total temperature is equal to the sum of these two, namely, *nf*.

Hence this general equation (26) for the temperature at any point shows that in any case the total temperature is the sum of the temperatures due to each of the two kinds of heat flowing by themselves; that is, *the heat gradient ordinates of the combined flow of heat are equal to the sums of those of the component flows of heat.* Thus the ordinates of the gradient P are the sum of those of the line cb (conduction alone) and those of the curve p (C^2R heat alone). If this is considered to be axiomatic, it may be taken as the starting point for deducing the other equations.

Returning to the general equation (26) for the heat gradient, it will be found that when half the C^2R heat W is made greater than H, the heat gradient becomes the parabola P'. This means that the electrode becomes hotter than the furnace at a point within the furnace walls; the heat gradient at the furnace end c then is downward toward the furnace, which means that some of this C^2R heat then flows into the furnace. The electrode is then too long; it ought to be shortened and have less C^2R loss, thereby economizing in both material and power.

If, on the other hand, half the C^2R heat W is made less than H, the heat gradient becomes the parabola P''. This means that the hot end of the electrode is cooler than the furnace, and therefore abstracts heat from it; the heat gradient at c now is downward away from the furnace. For the parabola P (minimum loss) the heat gradient at c is horizontal, which means that no heat either enters or leaves through the hot end.

It might be added here that if the heat conductivities k are known only for the two extreme temperatures, it is correct to use their arithmetic mean in the above formulas, except in the one for the heat gradient. This assumes that this conductivity varies according to a straight line law. This assumption, however, is not quite correct, but the error will then be only that due to the difference between the straight line law and the true one, which in most cases will probably be within the limits of accuracy of furnace calculations. But if the mean value is determined as above described, and then used in these formulas, the results ought to be correct and the errors ought to cancel each other, because the empirical value was then calculated with the same formula in which its value is afterwards used. To intro-

duce any other than a straight line law into the above equations would make them more cumbersome than the extra accuracy would seem to warrant.

There will be a slight difference in the heat gradient obtained when the conductivity is assumed to vary by a straight line law, and when a mean value is used, although the end results are the same. Fig. 9 shows the two curves; in *cPb* the conductivity is assumed to be constant (the mean value) for the whole range, and in *cP'b* its true value is used for each point.

In conclusion, the writer desires to repeat that there are, of course, many other considerations besides the mere power loss

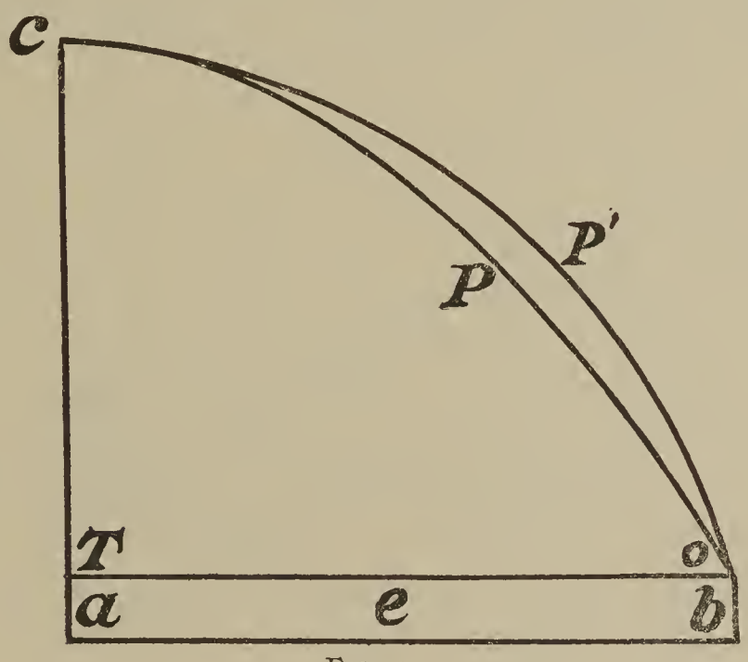


FIG. 9.

and economy of electrode material which should be taken into consideration in the design of electrodes, some of which may at times outweigh the latter in importance; this depends on the particular conditions of the case. It is the duty of the designer to properly weigh all the various factors; he can, however, do this intelligently only when he knows what the ideal results are; the intention of the present investigation was therefore to assist the designer by showing what the best results are as far as it concerns only two of the many factors, the size of the electrodes and the power loss in them; the intention was not to attach undue importance to these two factors over the others. If, for instance, the consumption of an electrode in an arc furnace

is in proportion to the quantity of electricity, it may perhaps be a distinct advantage to make them large so as to reduce the speed of feeding. If so, the above analysis will show that in such a case the length of the embedded electrode should be proportionately increased or else the power loss will become greater; it also enables one to determine what this increased power loss would be, and therefore whether it is worth saving.

Metric and Fahrenheit Units.

The inch and the centigrade scale were adopted in the above formulas because the former is the common unit of length used in this country and the latter is the temperature scale most commonly used in tables of references, in researches and in most pyrometers. When all the quantities are in terms of centimeters, gram calories and centigrade degrees, hence in absolute units (except the current), the constants in the above formulas (1) to (9) will be the same; that is, the formulas are equally applicable to either set of units provided those units are used consistently throughout. When T is in Fahrenheit degrees, and k is in terms of thermal units for one inch cube and one degree Fahrenheit, that is, pound-Fahrenheit units commonly represented by B.T.U., and if L, S, and r remain in inch units as originally specified, then the constant 4.186 in formula (1) becomes 1055.; the constant 2.894 in formulas (2), (3), (4) and (7) becomes 45.93; and the constant 0.3456 in formulas (5), (6), (8) and (9), becomes 0.02177.

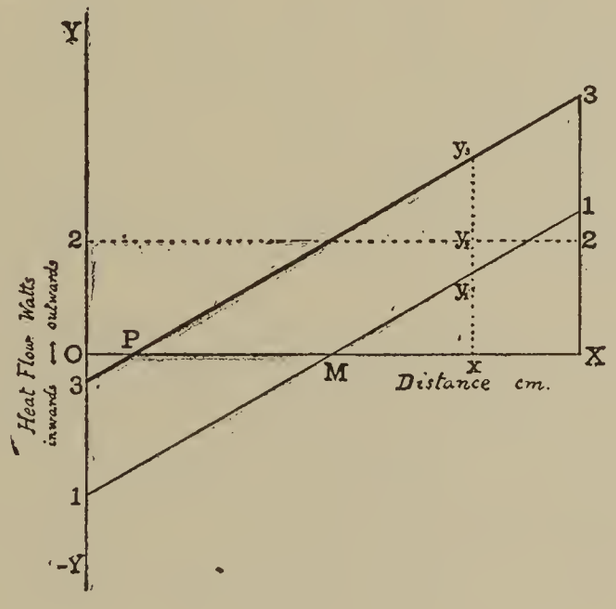
Philadelphia, June, 1909.

Note.—During the discussion of this paper, in the form in which it was presented at the meeting, Dr. Roeber, Dr. Richards and Mr. Arsem called attention to an inconsistency in that the algebraic proof is limited to constant conductivities, while the premises assumed that the heat conductivity may vary with the temperature according to a straight line law. Hence the proof does not embrace this feature in the premises. In this they are, of course, quite correct and I take pleasure in acknowledging my indebtedness to them for calling attention to it, and for their permission to change it in the paper as now printed by restricting

the premises, in order to make it consistent. The origin of this oversight and the effect which varying conductivities have on the results, will be referred to in my reply to the discussion.

DISCUSSION.

DR. A. E. KENNELLY *(Communicated)*: Whatever modifications or limitations may be imposed by practical conditions, there can be little doubt that Mr. Hering's paper expresses the main theoretical laws of economy in furnace electrodes. Curiously enough, there is a close analogy between the law here presented of the



size of electrode for minimum total heat waste and the Kelvin law of the size of a transmission conductor for minimum total annual cost. In each case we have the addition of two terms, one of which descends along a hyperbola, and the other rises uniformly along a straight line. The intersection of the two marks the point of theoretical maximum economy.

A few of the results which are arrived at in Mr. Hering's interesting paper may be illustrated in a somewhat different way, with the aid of a diagram containing only straight lines.

Let OX represent, to scale, the length of the furnace electrode, from the end O in the furnace to X the free outside end. M represents the middle point of the electrode length. Ordinates meas-

ured along the axis YOY represent heat flow along the electrode in watts. Positive heat flows, or ordinates above O, represent flow of heat outwards from the furnace; while negative ordinates represent flow of heat inwards towards the furnace.

We now consider three successive thermal states:

(1) *The furnace cold and the current flowing through the electrode.*

Let the furnace end O of the electrode be kept somehow at the same temperature as the outer end X, say, by means of a water ring at O. Let the full current strength be kept flowing through the electrode OX. If the electrode is thermally insulated at the sides, all of the I^2R heat will escape equally at the two ends, in the steady state. This condition is represented by the straight line 1M1. At X we have the flow X1 outward. At O we have the same number of watts O1 inwards. At the middle point M there is no flow in either direction. The mid-point M is the point of highest temperature. At any point x the flow is xy_1 watts. The temperature elevation of the point x over that of the extremity X is proportional to the area $xX1y_1$.

(2) *The furnace hot and the current shut off the electrode.*

Let the furnace be kept somehow at its full normal temperature elevation, and the end X kept at normal low temperature with the current shut off. Then, in the steady state, there will be a uniform heat flow along the electrode, as indicated by the horizontal dotted line 2, 2. The temperature elevation of the point x with respect to X is measured by the area $xX2y_2$.

(3) *The furnace hot and the current flowing through the electrode.*

In this final state, representing normal working conditions, the two preceding thermal states are superposed. We thus add corresponding ordinates on the lines 1,1, and 2,2, to produce the heavy line 3,3. Under the conditions shown, with the ordinate X2 less than X1, we obtain a small flow O3 watts into the furnace, and the flow X3 watts through the free end. P is the point of no flow, and of highest temperature. At any point x, the temperature elevation above X is measured by the area $xX3y_3$, the maximum temperature elevation being X3P.

The remarkable proposition of Mr. Hering's is that there will be the theoretical minimum of heat waste from the electrode when $X_1 = X_2 = (X_3)/2$; so that O becomes the point of highest temperature in the final state, with no flow at O.

MR. CARL HERING: Dr. Kennelly's diagram is a very ingenious and useful way of representing and studying some of these complex quantities, because there are no curves in it. On the other hand, a straight line diagram never shows any minimum values, and that is one of the very things we are often after. The other point against it is that temperatures are represented by areas, which is a somewhat awkward way to represent them. Whenever these two features are of no importance, this is a very ingenious way to represent these complicated relations.

PROF. JOS. W. RICHARDS: When Mr. Hering uses k and r as true of the electrode material in *any* differential of its length (as in formulas 10 and 11), he virtually uses these values as applying to every element of the length, and therefore to every temperature prevailing in the electrode. The mathematical result is a formula whose relations or nature are determined by, or virtually based upon, *constant* values of k and r for *all* temperatures. The formulæ are not mathematically correct if k and r vary with the temperature, as they do considerably. Mr. Hering calls k and r *mean* values, and thinks that he has assumed these to vary with temperature "according to a straight line law." The derivation of the formulæ, however, shows it not to admit of any such assumption, but to be true only if k and r are *constant for all temperatures.*

That this criticism of the inaccuracy of the fundamental formula is correct may be seen concretely in formula 22, where the equation of the parabola *p* of Fig. 8 is obtained by assuming zero temperature at the two ends of the electrode, and in which k and r are, by assumption or premise, the constant values between these two temperatures, *i. e.,* the values at 0°. Thus no provision is made in the formula for the great changes of *k* and *r* with increasing temperature towards the middle of the bar, the formula giving only the curve on the assumption of constant values of k and r, equal to their values at 0° throughout.

I have not evaluated the differences between the results of

Mr. Hering's formula and those obtained by using the variable conductivities (thermal and electrical), but my feeling is that the error of the formula as developed by Mr. Hering will be considerable. It gives us, however, a valuable first approximation in a question which is very difficult to solve.

MR HERING: I have anticipated this criticism in the paper by admitting very clearly that the conductivities vary somewhat with the temperature. It is clearly stated on the fourth page and referred to again in other parts. Prof. Richards is therefore very much mistaken when he states that I omitted mentioning this, or that I did not recognize it. Moreover, I stated in my verbal abstract at this meeting that what we should now do is to find out how nearly this is a safe approximation. I do not agree at all with Prof. Richards that the variation is likely to be so great as to "seriously vitiate the result." On the contrary, I believe the errors will be relatively small, and probably entirely negligible. I have stated clearly in the paper, and repeated it in my verbal abstract, that these constants come in the formulas in such a way that great accuracy in their values is not required. As a matter of fact, many of the electrodes in actual use are so very much at variance with the best proportions that we could in many cases get very much closer to the ideal conditions even though we do not know the constants accurately, and even though they are not quite constant.

I have stated in the paper that I assume the conductivities to vary according to a straight line law, which, of course, means a slanting line; and I have stated that I use the average value for this, just as it is generally done in similar cases. To introduce into this investigation a formula expressing the variation of that constant with the temperature makes it so difficult to solve that it is not worth while doing it. I tried to integrate these expressions when they include this variation factor, but the result would be of no use in practice—it is too complicated.

The conductivities which Prof. Richards has referred to do not increase with temperature. The heat conductivity of electrode materials used nowadays decreases as the temperature rises. That is correct, is it not, Mr. Hansen?

MR. HANSEN: I think so.

MR. HERING: The heat conductivity does not increase, but decreases.

PROF. RICHARDS: Whether the conductivities increase or decrease does not alter my point. Mr. Hering has stated in his reply exactly what I object to, that it is *assumed* that the heat conductivity varies with the temperature. He may think that he is assuming that, but he has *used* it as if it was a constant. His mathematical formula uses that quantity as if it is a constant, and is only true in the case it is a constant, independent of temperature.

MR. HERING: Prof. Richards' point is covered clearly in my Fig. 9, which anticipates the very question he raises. It is an academic refinement, not worth much consideration in practice. Fig. 9 shows the difference between the approximate and the strictly correct result. It shows that while there is a small difference in the middle of the electrode, the two end results come out correctly, and it is the end results that interest us in practice.

MR. W. C. ARSEM: I would point out what I think is a fundamental error in Dr. Hering's theory. His formula No. 11 contains a value k which is assumed to be the constant of heat conductivity. Dr. Richards has brought up the point that the heat conductivitity is not a constant, but varies with the temperature. Consequently, in Dr. Hering's formula No. 11, he should substitute for k its equivalent $k = ko\ (1 + at)$. The subsequent integration would undoubtedly give a quite different final equation for the total heat loss, so that it will not do merely to assume a mean heat conductivity of a certain magnitude, depending on the average temperature of the electrode, and substitute this value in the equation, because the equation is affected by the error in deduction which I have indicated.

MR. GEO. G. GROWER, Ansonia, Conn.: Some years ago I made an investigation. It had nothing to do with electric furnaces, but the conditions were similar, dealing with electrical conductors having their ends at different temperatures, and I arrived at substantially the same formula as Mr. Hering has done in this case. One way of arriving at the same result would be: If we imagined the electrodes as entirely immersed in the

furnace, it is very evident that the loss would be merely the thermal conduction loss. On the other hand, if we imagine the electrodes as removed entirely away from the furnace, the loss would be the sum of the thermal conduction loss and the C^2R loss; but as a matter of fact, the electrodes run from the outside to the inside, and therefore the loss, as Mr. Hering points out, is one-half the C^2R loss added to the normal thermal conduction loss.

Mr. Hering also shows that this simmers down to a certain relation of area and length. That is very true in a certain sense, but if we assume that thermal conductivities and electric conductivities are maintained at a constant ratio right through, the dimensions or the proportions are of no particular consequence if we can operate the furnace at a different activity. In other words, you can give these electrodes any proportions you may choose, and if you can run the activity of the furnace up or down to suit the size of the electrodes, you get exactly the same efficiency. In other words, the efficiency is a function of temperature and activity, and is independent of the dimensions of electrodes.

Just one other point. I do not think Mr. Hering means to suggest here in Fig. 1 that there is any practicable advantage in heating the electrode by the current up to a higher point than the temperature of the furnace. That would simply mean that although there might be no conduction from the furnace itself, the conduction comes from the highest point of the electrode to the lowest, and therefore the thermal loss is there, whether it comes from the inside of the furnace or not. And, again, there are two conditions which make this still worse, because the temperature is higher than in the furnace, therefore the thermal losses would be greater, and also the mere fact that this electrode is being heated to a higher temperature, short of its extreme length, would shorten the length of the conductor, and we would have increased loss in two ways, because we have a higher temperature and also because we have a shorter length.

Mr. Hering: The matter is stated in the paper as Mr. Grower has just explained it. Concerning Fig. 1, which seems to have been misunderstood repeatedly, I desire to explain again

that I used that exaggerated illustration merely because it is a good way to show what actually takes place. I said six months ago before this Society, and repeated it on the second page of this paper, that the hottest section will, of course, in practice be made the end of the electrode, and the temperature there be made approximately equal to that of the furnace.

Prof. J. W. Richards: I wish to repeat my former statement, that I believe that the fact that Mr. Hering's original equation is based (whether he says so or not) upon the *constancy* of the thermal and electrical conductivities, introduces an uncertainty into the formulæ as a whole, and therefore determining the end temperature and applying the formulæ to determine the mean conductivity or mean resistivity, will give fallacious results.

Mr. F. A. Lidbury: There is one point that I should like to see brought out in connection with the assumption to which Dr. Richards objected in Dr. Hering's analysis. The objection was that not only did the conductivities change with the temperature, but that their mean values also changed with the temperature. In an application like the present one, however, where the temperatures at the ends of the experimental electrode are precisely those which are to apply in the practical case that is being considered, these mean conductivities must have definite, invariable values. I would like to have Dr. Richards' and Dr. Hering's opinions on the question whether, under these circumstances, the application is not correct; since, if the results that can be obtained by measurements on an experimental electrode can be applied to operating conditions between the same temperature values, it is very important, and makes Dr. Hering's method of great value in actual practice. It is not so important whether the measurements taken at one temperature can be applied at another or not, since the actual temperature conditions will usually be reproducible.

Mr. Carl Hering: As stated in the paper, the formula for determining the constant by this method is exactly the same as the formula in which it is afterwards used; hence it is quite correct to use that value which is determined by this method, no matter what the variations are. It should be borne in mind, however, that it is not right to make this test for 3,000 degrees and then use that constant for a lower range of temperature. The

test should be made for the temperature of the furnace, whatever that is, and then I think the criticisms concerning the effect of the variability of the constant on the formulas, made in the discussion of my other paper, no longer hold. I acknowledged at the start that these analytical deductions are strictly correct only when the premises are strictly correct. I tried long ago to introduce the correction for varying conductivities. I originally tried the analysis in that way, and found that the integration was practically impossible, or if possible, the result would be so cumbersome that no one would think of using the formulas in practice. I concluded that the best way to get useful results was to use the simple conditions, and then make the necessary corrections afterwards, after having determined what they should be. If they are very large, the formulas become less useful, but if they are not large, they can readily be added as correctional factors, or more probably, dropped altogether.

Dr. Horace Clark Richards, University of Pennsylvania (*Communicated*): By the courtesy of Mr. Hering I am permitted to contribute a few words to the discussion of his paper. The interesting results which he obtained are on the assumption that the electrical and thermal conductivities of the electrode are constant. As the range of temperature is so great, it seemed desirable to consider the effect of variations of these quantities; and I have succeeded in obtaining a solution of the problem, which I hope shortly to publish elsewhere. The results of the investigation, as far as they bear on the present discussion, are as follows:

Let the thermal conductivity be given by the formula

$$k = k_0 (1 + a\,t + \ldots.)$$

and the electrical resistivity by a similar formula

$$r = r_0 (1 + a^1 t + \ldots.)$$

where k_0 and r_0 are the values for k and r at the cold end. (If only two terms of the series are used, a and a^1 are the temperature coefficients of k and r respectively.) Then the equation (5) of Mr. Hering's paper for the best proportions for the electrodes will be modified to

$$(5^1) \quad \frac{S}{L} = 0.3456\, C \sqrt{\frac{r_0}{k_0}\, T} \times \left(1 + \frac{5a^1 - 3a}{12}\, T + \ldots\right)$$

and the total loss in watts in this minimum case will be given by

$$(2^1) \quad mX = 2.894\, C \sqrt{k_0\, r_0\, T} \times \left(1 + \frac{a + a^1}{4} T + \ldots\right)$$

instead of equation (2) of the paper. In our present lack of knowledge as to the variation of k with temperature it does not seem worth while to write down the higher terms of the series.

It thus appears that the general conclusions, (c), (d), (e), (f) of the paper are valid even when the constants of the material vary with temperature; and while in this case the laws (a), (b) and (g) are not strictly true, the error will be small unless there are enormous changes with temperature. In fact, if it is remembered that the k and r of Mr. Hering's formulas are the *average* values (*e. g.*, $k = k_0\ (1 + \frac{1}{2}\, a\, T)$, it may be shown that to the first order the expression (2) for the watts lost agrees with the correct formula (2^1) above; while the value of S/L calculated from the approximate formula will be in error by the fraction $a^1T/6$—too large if a^1 is negative, *i. e.*, if the substance is, like carbon, a better conductor of electricity hot than cold. Taking a^1 as —0.0003, and T as 1700°, this would indicate an error of only 8½ per cent., which under the circumstances is insignificant.

Carl Hering *(Communicated)*: Summarizing this discussion briefly (including Dr. Roeber's paper) it seems that the analysis, laws and formulas are rigidly correct under the assumed premises, and that whatever errors there may be embodied in the premises, due to varying conductivities, are too small to be of any importance in practice, they being apparently smaller than the limits of accuracy with which a furnace can at present be designed and operated. All objections seem to be overcome when the complete method described in the paper is used, namely determining the true and correct mean conductivities by means of the same formulas in which they are afterwards used. Hence whatever doubts were raised concerning supposed errors, they do not apply to the complete method.

Coming now to details, the chief point raised against the paper in this discussion is that it was claimed that little faith can be had in these formulas, because I had for simplicity sake used the mean or average values of the conductivities, instead of the actual

values at every point along the electrode. Dr. J. W. Richards even went so far as to say that his "feeling is that the error of the formulas as developed by Mr. Hering will be considerable." He also "believes" that the formulas "will give fallacious results." This, however, was only a "feeling," "belief" or supposition on the part of my critics, as none of them could present any actual facts or figures to show that this error in the approximate premises was really of sufficient importance to warrant such a condemnation of the whole analysis. That corrections of the results would be necessary, if great accuracy was required, was clearly admitted in the paper, hence no claims were made that the calculated results would agree exactly with the actual ones. The only thing that was claimed in the paper was that the results are rigidly correct and not approximate, *under the assumed premises;* that is, it was the premises and not the mathematical deductions, which were only approximate, hence the laws given are absolute under those premises.

I admit that the rigid proof as given did not embrace the extension of the premises to include the case of the heat conductivity varying according to a straight line law, and with the permission of those who pointed this out, I have, in the revised paper as now printed, limited this premise to embrace the mean or average value only, so that it is now consistent throughout and is rigidly correct under the stated premises.

I do not, however, admit hereby that the statements as originally made were in error, as was claimed by Mr. Arsem; the facts are that they were quite correct. I merely admit that I had not given any proof in the paper, of the application of these laws to straight line variations of the heat conductivities. When the paper was originally prepared last May, I realized that the then unknown and therefore possibly great temperature variation in the heat conductivity, might give rise to a rather large correction. And as the rigid mathematical solution of this second approximation seemed too intricate to warrant spending the time over it then, I made calculations of actual cases in which great variations in the heat conductivity, according to a straight line law, were assumed, and the end results (see fig. 9 and the paragraphs preceding it), always came out the same as when calculated with the single, mean, average value; hence I felt con-

vinced that the laws did include this case, and that some general algebraic solution of it could be found if it was worth while to look for it. This rigid proof has now been supplied in the very interesting and able paper of Dr. Roeber published elsewhere in this volume. Hence the laws and formulas are quite correct even for broader premises than those to which they have now been limited. The criticism, as far as the variable heat conductivity is concerned (which was all that was included in the paper), therefore falls, the "supposed" error being exactly zero.

As the change in electrical conductivity has long been known to be only about 50 per cent., it was not thought necessary at that time to investigate the effect of this change on the formulas which are based on the mean value. Hence nothing was claimed in the premises concerning variations in the electrical resistivities. Dr. Kennelly has now shown (see below) that under slightly different premises the same is true of the electrical resistivity that Dr. Roeber has found to be true of the thermal conductivity, namely that it is correct to use the mean value if the variation follows a straight line law. It now seems that whatever small differences there may be between the approximate premises and the more accurate ones, are due to the mutual effect of both variations on each other, and not to the variation of either property alone. But as will be seen below, this error seems to be so small that it is entirely negligible in practice, when we remember that furnaces cannot at the present time be designed, constructed and operated under exact conditions.

The smallness of the error is apparently due to the fact to which attention was called in the paper, and emphasized in my verbal abstract at the meeting, that the minimum curve is quite flat at the minimum point. As there stated, an error of as much as 10 to 20 per cent. in the cross section or length makes an error (in the particular example worked out in the paper) of only 1 to 3 per cent. in the minimum loss, which of course is quite negligible in practice.

Electrodes are generally used in stock sizes, and while their lengths could be cut shorter, the cross section must generally be selected from the nearest standard size. Hence great accuracy in the proportions cannot be carried out in general practice. It

is fortunate, therefore, that the analysis shows that great accuracy in the dimensions is not at all necessary, as far as the loss of energy is concerned. The two important considerations in the dimensions, are the economy of material and avoiding an unnecessary excess of temperature within the furnace wall, arising from unnecessary length or too small a section.

Dr. Roeber's interesting solution is limited to the ratio of section to length, which is the less important of the two quantities, as far as accuracy is concerned, a relatively large error in that ratio being of small importance. It would of course be quite improper to conclude that the error in the more important quantity, the minimum loss, is equally as great as that found from his formulas; it will unquestionably be much smaller, probably not more than about a tenth, owing to the flatness of the minimum curve.

As to the uncertainty of the constants, I repeat that they both occur under the square root sign and the importance of accuracy is thereby reduced.

Mr. Lidbury has taken a very practical and sensible view of the matter (see his remarks in the discussion of my other paper). I tried to make clear in the paper the point which he now emphasizes; he seems to have been among the few who understood that point. The values which are used in my formulas are *not* independent of the temperatures, in any particular case, but on the contrary are very much dependent on it. The value for one range of temperatures would in my formulas be quite different from that for a different range, as it is in each case the mean or average for that particular range, as is clearly and repeatedly stated in the paper. His question is therefore answered as follows: If the mean, definite and invariable value is determined by experiment, using the same formula for calculating it as that in which this single value is afterwards used, and the same temperature ranges, the results will be correct; nobody has questioned this. This is the complete method which I suggested in the paper, but some of my critics seem to have overlooked this main point by looking with microscopes for mere academic differences.

Prof. J. W. Richards has gotten things somewhat mixed. I did not only "think" that I had assumed the average or mean

values to vary with the temperature ranges, but I even knew it and fully realized it; I therefore clearly stated that the mean value should be taken for the particular range of temperature of each given case. He is, therefore, entirely in the wrong if he claims broadly that the values of k and r in my paper are "constant for all temperatures." What he probably meant to say was that if in any particular problem a set of variable values is for simplicity sake represented by their average, then that average is a constant in that problem and some of the individual values necessarily differ somewhat from this average. As this is always the case in all such very common every-day problems in which average values are used, I assumed it to be generally known and therefore hoped it would not be necessary to describe it in detail, but I consider that my paper shows that I realized it.

The extremes to which one of the critics went in endeavoring to discredit the results, is shown by the statement that the alleged "inaccuracy of the fundamental formula" is "concretely" proved by formula 22. The average values of k and r refer throughout the whole paper to the mean for the range from the highest to the lowest temperatures. This is clearly and repeatedly stated. Moreover, it was also clearly shown that the highest temperature in that exceptional case of formula (22) was in the middle; hence the range of temperature in that particular case is of course understood to be that between the middle and the ends and not that between the two ends (which are now alike) as Prof. J. W. Richards has supposed was meant. If there is any doubt about what my meaning was, as to the mean value, my second paper on a method of determining this, which method is based on this very case of formula 22, ought to have made this clear. My reply to this criticism, therefore, is that the mean values of k and r in this special case *do* provide very effectively for the variations due to different temperature ranges, and that it was for this very reason that my proposed method of determining this correct average for any given range, was based on this very case. Moreover, no one else has questioned this.

That there is no "fundamental error" in the paper, as claimed by Mr. Arsem, is shown above.

From the practical standpoint the most valuable parts of the

discussion are the results obtained by Dr. H. C. Richards, and those by Dr. A. E. Kennelly referred to below.

Dr. H. C. Richards' interesting formula gives the second term of the integration and hence is a second approximation, beyond which it would probably never be necessary to go in practice. It, therefore, makes it possible to answer the unsupported claims or insinuations made by some of the critics, that the errors due to the varying conductivities, are important or large.

To show this with actual figures, I have calculated the results obtained from these more exact formulas and compared them with those obtained from the very much simpler ones given in the paper. This has been limited to graphite because carbon may become partially graphitized and therefore be an unknown mixture having doubtful constants. The data used is the same as that in the numerical example in the paper; it is now assumed that both the electrical resistivity r and the thermal conductivity k are half as large at the hot end as at the cold, that they vary according to a straight line law, and that the mean values are those given in the paper. This variation is about correct for the resistivity, which we have known for many years to be about half as great when hot than when cold. As to the heat conductivity, nothing much is known except Mr. Hansen's general result that it diminishes with temperature. It is here assumed to vary by 50 per cent. also.

The results are as follows: If R and r are the electrical resistivities, cold and hot respectively, and K and k the thermal conductivities, cold and hot, then in Dr. H. C. Richards' formulas $k = K(1 + aT)$, $r = R(1 + a'T)$ the values of a and a' will both be -0.0003125. Moreover $k = 0.16$, $K = 0.32$, $r = 0.0001773$, and $R = 0.0003545$.

Substituting in Dr. H. C. Richards' formula for S/L gives 2.64, while using the mean values $k = 0.24$ and $r = 0.000266$ in the simple formula (5) in the paper, gives 2.88, an error of only 9.1 per cent., which of course is neglibly small in practice. But the minimum loss is by far the more important of the two results; substituting in his formula for mX, gives 9.247 k.w. as against 9.240 obtained from the simple formula, thus showing practically absolute agreement; even this small difference may be due only to the arithmetic figuring.

Let it be assumed now that the thermal conductivity varied as much as 10 to 1, the resistivity variation remaining the same, namely 2 to 1. Then a becomes —0.0005625, $k = 0.04364$ and $K = 0.4364$. Then S/L becomes 2.50, which means an error of only 15 per cent. The minimum loss then is 9.358 kw., showing the error of the approximate formula to be only 1¼ per cent., which again is absolutely negligible in practice.

This ought to settle the doubt which has unfortunately been raised by those who "supposed" or "felt" that the error was so large that it vitiated the formulas. It shows that a mountain had been made of a mole hill.

In laying so much stress on the supposed error due to varying conductivities, what may be the larger error, was entirely overlooked by the critics, namely the one in the other premise concerning perfect heat insulation, which has been assumed in all the other later analyses of the more complete case. The quantity of heat leaving the electrode within the walls does not come under the square root sign (like the physical properties do) nor is it affected by the flatness of the minimum curve and hence it seems to me that the attacks had better have been directed to it, instead of to the almost microscopic temperature corrections.

It is of interest to note from the above more accurate figures that whatever error there may be is on the safe side, as the cross section calculated from the approximate formula becomes slightly larger (or the length slightly shorter) than is necessary. The other error is too small to be considered at all.

Dr. Kennelly attacked the solution of the general problem in a different way, and with his permission I am able to give his very interesting general results here; his complete paper on this subject will be published elsewhere, and is recommended to those interested in the analyses.

He discusses the laws a and b of my paper with a view of finding how far the temperature variations of the conductivities modify them. He uses the resistivities for both the electric and the thermic properties, and assumes them both to be known for the two extreme temperatures; also that the variation of the resistivities follows a straight line law; also that the temperature increases uniformly from the cold to the hot end, although acknowledging that this is only approximately correct. Under

these approximate premises he finds by the aid of the calculus a number of interesting and valuable results among which are the following:

"The thermal resistance of the electrode with a thermal resistivity varying in the manner assumed, is the same as that of an electrode having the same dimensions and a constant resistivity, equal to the arithmetic mean restivity of the first.

"The total resistance of the electrode with an electric resistivity varying in the manner assumed, is the same as that of an electrode having the same dimensions and a constant resistivity equal to the arithmetic mean resistivity of the first.

"The electric resistivity alone varying, the joulean heat does not divide equally between two ends of the electrode.

"In the general case when both resistivities vary, the minimum heat waste is found when the joulean flow through the external end is equal to the furnace flow." But he adds, that the portion of the joulean heat which flows out through the external end is not in general half of the total joulean heat when either one or both of the resistivities can vary.

"Within the assumptions to which the above inquiry has been confined, Hering's first two laws may be amended as follows to meet the conditions of variable thermal and electric resistivity:

"a. The combined loss through the cold end of an electrode is equivalent to the sum of the loss by heat conduction alone (when there is no current) and approximately half the I^2R loss, the exact fraction depending on the temperature coefficients of the resistivities. With constant resistivities the fraction will be ½.

"b. This combined loss will be least when the loss by heat conduction alone is equal to the joulean loss through the cold end. The total loss will then be approximately equal to the total joulean loss and very little heat will flow into or out of the furnace. With constant resistivities this resultant furnace flow will be nil."

It may be of interest to call attention here to the fact that in assuming straight line laws of variation with the temperature, Dr. Roeber applies this law to the two conductivities, Dr. Kennelly to the two resistivities, and Dr. H. C. Richards to the electrical resistivity and the thermal conductivity, the latter being evidently done to conform with the original paper, in which I used this hybrid combination only because those are the units com-

monly used in practice, the formulas being intended for direct practical application; in my cases the analysis was merely incidental to an effort to get at some better practical result than our former rules gave us. As the resistivity and the conductivity are reciprocals of each other, it necessarily follows that if one varies according to a straight line law the other does not. Hence exact agreement cannot be expected in these three interesting solutions. While this will make no difference in the results as far as their practical application is concerned, it might make just enough difference to complicate an exact mathematical comparison. It was in anticipation of all these and other complications that my suggested solution of the complete problem was based on measuring the correct mean value with the aid of the same formula in which it was afterwards used.

The reason for using average values for the conductivities in my formulas, was not only because I considered this to be a close enough approximation for all practical work, at least for the present, but also because those simple formulas enable other interesting deductions to be made, which might not have suggested themselves if the original formulas had been burdened with the complications of academic precision for the most involved case. I therefore am still of the opinion that in developing theoretical relations on new lines, one ought to start first with the simplest possible premises for the first approximations and then let the second and any further approximations follow later.

Concerning the statements and insinuations made at the meeting and since then, that my analysis, formulas and method of attacking the solution of this problem were old, well known, self-evident and obvious, I desire to say a few words in reply. There are many things that seem "obvious" to us *after* they have been shown us and proved, and it is then often possible and easy to find such a simple short-cut to the demonstration of the new results, that they seem to us quite self-evident and obvious, even though the original proof may have been complicated. But to therefore belittle the original work seems hardly fair and just. That my method of attacking this problem was considered to be little short of heresy and absurd only as recently as last May, was shown at our meeting in that month, at which I gave the brief outline and fundamental principles of it (see Vol XV, p.

300-302; the Figs. 1, 4 and 5 of my present paper were sketched on the blackboard at the time almost exactly as now reproduced, and Fig. 3 was described; but in view of the unfavorable reception of my remarks at that time, I did not have them reproduced in that discussion). The spirit in which it was then received showed that it was considered absurd or visionary; one of our ablest furnace experts said to me afterwards, "I hope you did not mean what you said." In view of the rule then in common use, namely, to reduce the electrical resistance of the electrodes as much as possible, I admit that it was not surprising to consider it little short of heresy to intentionally increase this resistance in order to reduce the total loss. My suggestion to raise the furnace end to the furnace temperature with C^2R heat, was apparently then considered an absurd waste of power. Now, the contrary is "obvious." Prior to that meeting the general rules for proportioning electrodes were based on lowest resistance, current densities, and the summation of the two losses. Now, it is "obvious" and "self-evident" that none of these rules are right, and that they were little short of crude guesses. Ever since that May meeting papers have been published in which these rules have been reiterated. At that meeting surprise was expressed at the then admittedly inexplainable result that the total loss in a certain case was even less than the C^2R loss; now it is "obvious" and "self-evident" that this necessarily results when the electrode is too long or the section too small. Before that May meeting, it was commonly accepted that graphite electrodes "chilled" the furnace on account of their high heat conductivity; now it is "evident" and "obvious" that electrodes might even be made of silver or copper, the best known heat conductors, without chilling the furnace a single degree.

It is very easy to say now that all this is simple and self-evident, as it undoubtedly is when looked at in the present light, but it ought not to be forgotten now that when the original announcement of the present "obvious" method of getting at the results was made last May, there were serious prejudices to overcome. Even prior to that May meeting I tried to interest one of the above critics in this new theory, more particularly in the important result concerning the constancy of the ratio of S to L to minimum conditions, but it failed to arouse the slightest interest

whatsoever. Now all this is "obvious." Now that the foundation of a rational method has been laid, it is hoped that the details and applications of this method will be further developed.

I wish to express my appreciation here of the very interesting and valuable investigations of Dr. Roeber, Dr. H. C. Richards and Dr. A. E. Kennelly, concerning the second approximation, or solutions of the general case. My replies to the papers of Dr. Roeber and Mr. Hansen are given in the discussions of those papers.

APPENDIX.

Since the above was written the writer has conducted a series of tests to determine with care the absolute values of the two conductivities required in the formulas given in the above paper. The method used is the one described in the paper following this one.

Up to the time of going to press with the present volume the range of temperature within which the values were determined by the writer, was from about 300° to 900° C.; beyond this temperature the rods were injured apparently by the refractory material used, or something contained in it. But the values within this range were determined with care and precision, and, therefore, may be used to at least indicate what the probable ones would be for higher temperatures. In general, the curves have but a slight slope at the highest temperature of the test (about 900° C.) and all appear to tend toward becoming nearly horizontal. Hence the error would probably not be great to use these values even for somewhat higher temperatures, until the actual determinations for these temperaturs have been made. This would surely be far more correct than to use the older data.

Reduced to a terminal temperature of 100° C., the values for about 900° furnace heat are as follows in the inch units specified in the paper: Graphite $r = 0.000323$; k 0.740. Carbon $r = 0.00148$; $k = 0.329$. They are presumably accurate to within a few percent, and were determined from a number of readings taken with considerable care.

For graphite, the electrical resistivity falls nearly rectilinearly from about 0.000330 at 390°, hence only slightly, the curve being nearly horizontal. The thermal conductivity falls faster, also nearly rectilinearly, being about 0.861 at 390°, but it seems also to tend to approach the horizontal.

For carbon, the electrical resistivity falls faster, from about 0.00166 at 360°; the curve is slightly concave upwards and tends to become horizontal. The thermal conductivity rises quite fast from about 0.226 at 360° and also tends rapidly to the horizontal, being concave downwards.

The much discussed ratio of the heat conductivities of graphite and carbon, therefore, turns out to be only about 2 to 1 instead of 8 to 1, 10 to 1 and 18 to 1, as have been claimed by others. This, at least, is the

case for 900° C. furnace heat, and applies to the mean values under operating electrode conditions, and when the electrodes are properly proportioned.

As these constants were determined by the method described, all questions concerning the corrections due to temperature coefficients, are eliminated. The values are in the proper form to use in the writer's formulas, under the assumption that there is no loss of furnace heat thru the electrode, which is the fundamental principle on which the writer's analysis was based.

It is of interest to notice that according to these constants for 900°, which may be relied upon under the assumption made, the so-called "electrode voltage" for one degree, for graphite, is 0.0447, and for carbon 0.0639. This means that the minimum loss for carbon is about 50 percent greater than for graphite, and not slightly less, as was found from the rough approximate constants used in the paper.

On the other hand, the difference in cross sections between carbons and graphites, for the same furnace and electrode length, is less than that determined from the older approximate figures, the carbons being now only 3.21 times as large as the graphites.

There is another matter of interest brought out by these tests. The mean electrical resistivity (under electrode conditions) falls more rapidly for carbon than for graphite. This means that an appreciably larger half of the heat due to electrical resistances will be generated in the colder half of the electrode, hence nearer the terminal. This heat will, therefore, have a shorter path for escape to the terminal, than if the heat were generated more evenly thruout, as with graphite. In other words, a carbon electrode, it seems, would be hotter a short distance from the cold terminal than one of graphite would. This, of course, is an unfavorable feature, and may account in part for the increased minimum loss for carbon. It seems to explain why the "mean" thermal conductivity turns out to be so unexpectedly high.

This difference in the distribution of the resistance heat along the electrode due to the change in electrical resistivity, shows that the mean value of the thermal conductivity found in this test and to be used in the formulas, is a specific kind of a mean peculiar to electrodes and might, therefore, be termed the "electrode mean" or the "mean under electrode conditions." It is neither an arithmetic nor a geometric mean, and will depend on the temperature coefficient of the electrical resistivity, and especially on its sign. This mean value of the thermal conductivity would, for instance, be different for carbon if the electrical resistivity rose with the temperature instead of falling. Hence it is not proper to compare these electrode mean values directly with the values given in tables for ordinary thermal conduction. The ordinary heat conductivity of carbon is certainly considerably less than that of iron, yet their electrode means are not very different, because the one has a negative and the other a positive temperature coefficient for the electrical resistivity.

The writer expects to publish the detailed results of these tests soon.

Philadelphia, January 19, 1910.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909; President L. H. Baekeland in the Chair.

A NEW METHOD OF MEASURING MEAN THERMAL AND ELECTRICAL CONDUCTIVITIES OF FURNACE ELECTRODES.

By Carl Hering.

The more extended introduction of electric furnaces and the generally high cost of electrical power, make it desirable to pay more attention to the design and proportioning of the parts so as to economize power, material, size, cost, etc., as much as practicable. Among the losses of power which are sometimes quite great, are the heat escaping through the walls of the furnaces and the electrodes, which latter are generally good heat conductors; also the resistance losses in the electrodes.

It has been shown in other articles and papers by the writer[1] how these heat losses can be calculated and how they can be reduced to a minimum by proper proportioning, provided the electrical and thermal conductivities of the materials are known, not only relatively but in absolute measures. It is therefore becoming important, in the present state of the art, to know what the values of these physical constants are; they have been determined only roughly, or relatively and in some cases not at all, or only for much lower ranges of temperature than those occurring in electric furnaces, and as these properties seem to vary very greatly with the temperature, particularly when it is quite high, the data for the lower ranges may lead one to erroneous proportions. The determinations of these physical properties for the ranges of electric furnace temperatures therefore is becoming important.

The object of the present paper is to suggest a new method applicable to electrical conductors as distinguished from the refractory heat insulators, and intended chiefly for the materials

[1] Heat Conductances Through Walls of Furnaces, Trans. Am. Electrochem. Soc., 14 (1908), p. 215. The Laws of Electrode Losses in Electric Furnaces, read at this meeting and published in part in Electrochem. and Met. Industry, October, 1909. Heat Conductance and Resistance of Composite Bodies, Ibid. January, 1909, p. 11.

used for electrodes. It has the advantage of simplicity and it is believed to be accurate, as there seem to be no sources of relatively large or uncertain errors. The basis of the method is a formula recently deduced by the writer for the relations between the temperature and the flow of heat under certain conditions.

Description of Test.

Let *ab* Fig. 1, be a rod of the electrode material to be tested, embedded in a heat insulating material *I*, except at its ends, which are water jacketed or otherwise cooled and kept at a constant known relatively low temperature. The refractory material *I* is assumed to be a perfect heat insulator; how to make it very

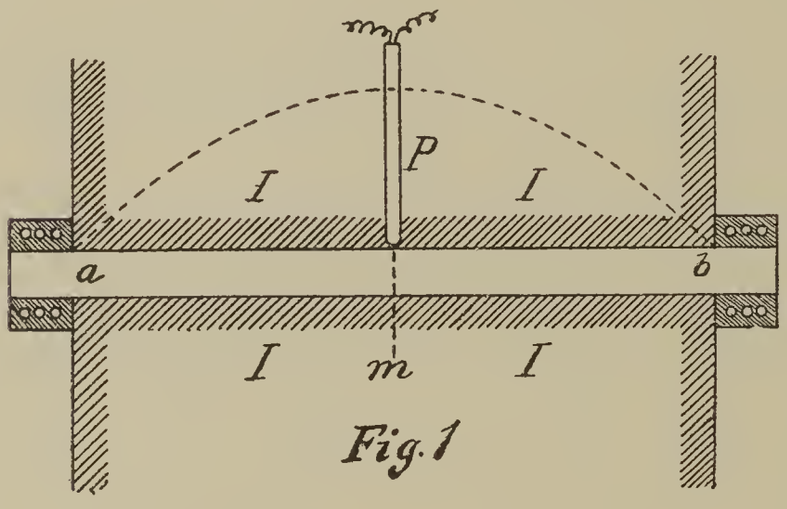


Fig. 1

Fig. 2

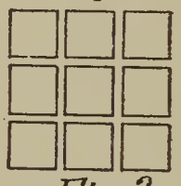


Fig. 3

nearly so, will be described later. The cross section of the rod and the length of the embedded part should be known.

Let a relatively large electric current be passed through this rod from end to end. This will raise the temperature of the embedded part, the highest being at the middle section *m*, and it will fall toward the two ends down to the temperature of the water cooled ends. It can be shown that under perfect conditions this heat gradient will be a parabola as indicated in dotted lines. A thermometer or pyrometer *P* is introduced at the middle point.

When the constant current has been passed for a sufficient length of time so that the stable state is reached, that is, until the pyrometer ceases to rise, then it is evident that all the heat generated in the rod electrically, flows out only at the two ends,

as the heat insulation is assumed to be perfect. When this state has been reached, the temperature at the center and at the water cooled ends, the current, and the voltage between the two terminals, should be measured. The product of the current and the voltage gives the heat flow in watts which is leaving at the two terminals. It will be noticed that it is not necessary to measure the heat absorbed by the cooling water, as this heat flow is determined much more readily and probably far more accurately by means of an ammeter and voltmeter.

The heat conductivity of this material is then determined from the following formula:

$$k = \frac{W\,L}{8\,j\,T\,S} \qquad (1)$$

in which k is the mean heat conductivity in gram calories per second for one degree C in cubic inch units; W, the watts in the whole rod, that is, the product of the current and the voltage; L, the length of the embedded part in inches; S, the cross section of the rod in square inches; T, the difference in temperature in centigrade degrees, between the maximum at the middle, and that at the water cooled ends; and j the electrical equivalent of heat, 4.186, by which the gram calories must be multiplied to reduce them to watts. Reduced to its simplest form this formula becomes

$$k = 0.02986\,\frac{W\,L}{T\,S} \qquad (2)$$

In designing furnaces it would be sufficiently accurate for most practical purposes to reduce this coefficient to the simpler number 0.03, the error being only about half of 1 per cent. It will be noticed that this value of k is the mean for that range of temperature, in just the form in which it is wanted for calculating the proportions of electrodes, and the losses in them. It is therefore not necessary to know the exact law of variation with the temperature, when the average value thus obtained is afterwards used in a similar formula for calculating electrodes.

Any desired high temperature can be obtained by increasing the current until that temperature is reached. The test can there-

fore readily be repeated for different ranges of temperatures. The current may of course be either direct or alternating, the former being preferable because the voltage will generally be low and instruments for measuring such voltages are more easily obtainable for direct than for alternating currents.

Heat Insulation.

This method is based on the assumption that the heat insulation of the test rod is perfect. This may be accomplished with probably no appreciable error by surrounding the test rod by a tube of the same or a similar material, which is electrically insulated from it, and which is also heated by a current to the same temperature at its middle part and cooled to the same temperature at the ends. The two heat gradients in the test rod and in the surrounding tube will then be the same, hence no heat will leave the embedded part of the test rod except the very slight amount which flows longitudinally through the cylindrical shell of heat insulating (and therefore non-conducting) material surrounding the rod and escapes at its outer ends. If this shell is made very thin, just thick enough to insulate electrically, and the rod be made relatively large in cross section, and long, the heat which is thus lost through this insulating material may be made extremely small, especially if the material is loosely fibrous or granular so that the actual heat insulation is practically due to the air in the interstices. Moreover only one-half of this small heat loss comes from the test rod, the other half coming from the tube.

Instead of using a tube for obtaining this heat insulation, which may at times be difficult to do, it is much simpler and probably quite sufficiently accurate for all practical purposes, to surround the test rod with a number of similar rods embedded in the same insulating material and placed parallel and as near as possible to the test rod about as shown in Figs. 2 and 3 in which the middle one is the rod under test. The ends of each are water jacketed like the test rod. These can then all be connected electrically in series with each other and with the test rod so that the same current heats them all; if then they are all of the same size and material, the temperature can be safely assumed to be practically the same in them as in the test rod, and it is then

unnecessary to use a second pyrometer on the outer row of rods to see that the temperature is the same as that of the test rod.

Electrical Resistivity.

From the same readings of the volts and amperes in this test the mean electrical resistivity for that same range of temperature is readily calculated from the formula

$$r = \frac{E\ S}{C\ L} \qquad (3)$$

in which r is the resistivity in ohm, cubic inch units, C is the current, E the voltage, L and S being, as before, the length and cross section in inches. It will be noticed that this also gives the same mean value for that range of temperature as is required to be used in subsequent calculations of electrodes, and that it is therefore not necessary to know the law of variation nor how to obtain the proper mean from it.

Advantages of the Method.

Among the advantages of this method are the following: It is simple, rapid, and requires no expensive or elaborate apparatus. It is not necessary to make any measurements of heat flows, as for instance by means of a measured flow of water whose temperatures before and after are measured, which is much more cumbersome and probably far less accurate than measuring the heat flow electrically in terms of the amperes and volts. The method is probably more accurate than others as there are no large sources of errors and uncertainties to allow for, because the heat insulation is nearly perfect. The test pieces are in the form of rods, either round or square, which are readily procurable in electrode materials and do not have to be especially made. The cross section and length could be selected to adapt the test to the current at one's disposal. The law of variation of the conductivity with temperature need not be determined; and the figure obtained for the conductivity is the proper mean value to use in the formulas for determining the proper proportions of electrodes and the loss in them regardless of what this law of variation is, provided only that the formulas are used consistently throughout;

hence such calculations do not involve any questionable assumptions as to what would be the proper one to use; in other words, the mean conductivity is determined under exactly similar conditions to those under which it is subsequently used, thus eliminating the knowledge of what the law of variation is or what mean is the proper one to use. The same readings taken for determining the mean heat conductivity also enable one to determine the mean electrical resistivity; and this mean value is also the proper mean to use in subsequent calculations of electrodes.

Illustration.

The writer had intended accompanying this paper with the results of a series of determinations of the two mean conductivities of the usual electrode materials carbon and graphite, but time did not permit making the tests before this meeting. The following illustration, based on approximate values, will, however, serve to give an idea of the quantities and dimensions involved.

Let the test rod be of graphite 12 inches long and ¾-inch diameter (cross section 0.442 sq. in.), and let 10 inches of it be embedded. Let the desired high temperature be 1700° C. and the low one at the water jacket, 100°, hence the drop in temperature will be 1600°. Suppose the test shows that it takes 307 amperes to bring the temperature up to the desired 1700°; and let the voltage between the terminals be found to be 1.85.

Substituting these values in formula (1) or (2), remembering that T is the drop in temperature, 1600°, and not the high temperature itself, gives $k = .24$ gram calories per second, in inch units. Substituting them in formula (3) gives $r = .000266$ ohms in inch units. These would therefore be the mean values for the range from 100° to 1700°, according to those readings.

Electrode Voltage.

There is a curious and interesting relation to which attention may here be called, as it bears on the voltages required for such tests. For a given temperature drop, this voltage between the two cold ends is always the same for any given material, no matter what the length or cross section of the sample is. A long or

short, thin or thick rod of a given material will require the same voltage to raise it to the same temperature at its middle point, assuming, of course, that the conditions are perfect. With less than this voltage it is impossible to raise the rod to that temperature and with a greater voltage the temperature will always be higher; no changes in the dimensions of the sample will change it. The current will be different, but even it will be the same for different lengths and cross sections when their ratio is the same. The current will, of course, be less the greater the proportion of the length to the cross section.

This voltage is different for different materials, and it is, therefore, quite as characteristic of the material as the thermal conductivity and the electrical resistivity are; for one degree drop in temperature it is numerically equal to 5.788 times the square root of their product (when the units are those stated above), the coefficient 5.788 being a mere mathematical constant involving no physical properties. This voltage may be said to be a physical property which depends on these two properties jointly, and as it has an important bearing in the selection of the material for electrodes of electrical furnaces, the writer has suggested the name "electrode voltage" for it.

The rod in this test may be said to represent the two electrodes of a furnace, the interior of the furnace being in this case a mere thin section through the middle point *m* in Fig. 1; hence this voltage is double what it would be for one electrode and the term "electrode voltage" is therefore best applied to one-half of it. The electrode voltage e of any material is therefore given by the formula

$$e = 2.894 \sqrt{kr} \qquad (4)$$

This is for one degree drop in temperature. It increases as the square root of the temperature drop, hence for any given range of temperature it is

$$e = 2.894 \sqrt{krT} \qquad (5)$$

For graphite the electrode voltage is therefore 0.0231, if the above values for the conductivity and resistivity are correct. It means that for any graphite electrode, no matter how large or small the furnace, the drop of voltage will be 0.0231 multiplied

by the square root of the temperature drop, assuming, of course, perfect conditions and the theoretically best proportions; and it will be the same whether the electrodes are large or small. This is further described in the writer's article above referred to on the Laws of Electrode Losses.

Deduction of the Formula.

The fundamental formula (1) is readily deduced from one of those given in the writer's paper above referred to on the Laws on Electrode Losses, and deduced therein. That formula gives the temperature to which the end of an electrode will be raised over that at the cold end, by the current flowing through it, provided no additional heat enters the electrode from the furnace (which is the case when this temperature of the electrode is equal to that in the furnace), and provided the electrode is heat insulated so that all the C^2R heat flows out only at the cold end.

That formula is

$$T = \frac{W\,L}{2jkS} \qquad (6)$$

in which the letters represent the same quantities as defined above, except that W and L now refer to only one-half of the corresponding quantities in Fig. 1; T refers to the drop from *m* to either end.

In the present case shown in Fig. 1, the single rod represents two electrodes with their hot ends butting together at *m*. Hence the measured watts and the length in Fig. 1 are each twice as great as the corresponding quantities in (6) and must therefore each be divided by two; this makes the coefficient in the denominator 8 instead of 2, and formula (6) then becomes formula (1), after solving for k.

Incidentally it may be of interest to notice that this temperature drop is just half that which would have to exist between the two ends of an electrode, to produce this same flow of heat at the cold end, when all the heat comes from the furnace, none of it being generated in the electrode.

Philadelphia, September 13, 1909.

DISCUSSION.

PROF. J. W. RICHARDS: I wish to repeat my former statement, that I believe that the fact that Mr. Hering's original equation is based (whether he says so or not) upon the *constancy* of the thermal and electrical conductivities, introduces an uncertainty into the formulæ as a whole, and therefore determining the end temperature and applying the formulæ to determine the mean conductivity or mean resistivity, will give fallacious results.

MR. F. A. LIBDURY: There is one point that I should like to see brought out in connection with the assumption to which Dr. Richards objected in Dr. Hering's analysis. The objection was that not only did the conductivities change with the temperature, but that their mean values also changed with the temperature. In an application like the present one, however, where the temperatures at the ends of the experimental electrode are precisely those which are to apply in the practical case that is being considered, these mean conductivities must have definite, invariable values. I would like to have Dr. Richards' and Dr. Hering's opinions on the question whether, under these circumstances, the application is not correct; since, if the results that can be obtained by measurements on an experimental electrode can be applied to operating conditions between the same temperature values, it is very important, and makes Dr. Hering's method of great value in actual practice. It is not so important whether the measurements taken at one temperature can be applied at another or not, since the actual temperature conditions will usually be reproducible.

MR. CARL HERING: As stated in the paper, the formula for determining the constant by this method is exactly the same as the formula in which it is afterwards used; hence it is quite correct to use that value which is determined by this method, no matter what the variations are. It should be borne in mind, however, that it is not right to make this test for 3,000 degrees and then use that constant for a lower range of temperature. The test should be made for the temperature of the furnace, whatever that is, and then I think the criticisms concerning the effect of the variability of the constant on the formulas, made in the discussion of my other paper, no longer hold. I acknowledged at the start that these analytical deductions are strictly correct only

when the premises are strictly correct. I tried long ago to introduce the correction for varying conductivities. I originally tried the analysis in that way, and found that the integration was practically impossible, or if possible, the result would be so cumbersome that no one would think of using the formulas in practice. I concluded that the best way to get useful results was to use the simple conditions, and then make the necessary corrections afterwards, after having determined what they should be. If they are very large, the formulas become less useful, but if they are not large, they can readily be added as correctional factors, or more probably, dropped altogether.

CARL HERING *(Communicated)*: The above remarks of Prof. J. W. Richards and Mr. Lidbury have also been answered in the closing discussion of my other paper. The only other discussion of the present paper is the comment of Dr. Roeber in a footnote of his paper, printed elsewhere in this volume.

Replying to the latter, he is of course correct in saying that there would be a difference of potential between the parallel rods, and hence a small leakage of current from rod to rod through the refractory material when it gets hot enough to become a conductor. My present belief is, however, that this leakage is only of academic and not of practical interest. As the "electrode voltage" of such materials at the usual furnace temperatures, appears from the best figures obtainable at present to be of the order of one volt, and probably less, the greatest possible difference in potential between two hot parts would not exceed about 12 volts when six heating rods surround the test rod, which is so low that the leakage current would probably be quite negligible, especially as this can again be cut in half by so connecting the rods that the test rod is electrically in the middle of the circuit. The error due to this leakage, if at all appreciable, could be further reduced by connecting the ammeter into the circuit at one end of the test rod, so that this leakage cross current would not enter into the formula. Our present knowledge of the electrical conductivity of refractory heat insulating materials is so meager that even an estimation of this supposed error is hardly possible. For exceedingly high temperatures, however, this cross current could probably be eliminated almost entirely by using air insula-

tion, with occasional thin disks to prevent circulation and direct radiation.

Connecting all seven electrodes in parallel and inserting the ammeter in the circuit of the middle one only, is of course an academically correct solution, but it is so much more cumbersome that it hardly seems worth while, as great accuracy in these constants is not required. It would involve the doubtful assumption that the current would divide equally among the six or eight auxiliary rods, and would rather require either a second ammeter for very large currents, or a second pyrometer and a rheostat to adjust this large current.

Although it is not touched upon in this discussion, I desire to add that it seems, at present at least, that no error, or at least no important one, is involved in making this test with relatively small rods and then applying the constants thus obtained to a larger one with a different current. The ratio of section to length, per ampere, it seems ought to be the same for all sizes, provided the conditions of no loss of furnace heat and the temperature ranges are the same in the electrode to be calculated as they are in the test. Moreover as both the graphite and carbon used are artificial products, and not chemical elements like the metals, absolute constancy in their physical constants is hardly to be expected, hence academic accuracy in their determination is hardly warranted; the constants for the test samples may differ appreciably from those of other lots.

In view of the academic criticism of my other paper, it might be added here that in the present test the supposed condition of perfect heat insulation is more nearly realized than in electrodes in actual practice. If this should trouble the minds of the academicians who are looking for minute errors, I might add that conditions which are slightly more nearly parallel to those in practice might be realized in this test, if instead of the surrounding auxiliary rods, the test rod were surrounded by a thin disk or flat coil, say of platinum, perpendicular to the rod at its middle point only, and heated to the same temperature as the middle section of the rod.

In the discussion of my other paper one critic interpreted the formula on which this test is based, as giving the conductivities at the cold end temperature only, and not as the mean for the

whole range. I wish therefore to explain, what I thought was self-evident from the repeated statements in the other paper, that the values obtained in this test are the means or averages for the specific ranges of temperatures of the test, and not those for the cold temperature; and that it was for this very reason that this test was suggested, as the electrical conductivity for ordinary temperatures can be determined without such a test, and it would be absurd to refer to determination of a heat conductivity between two points of equal temperature.

It might also be added here that for rough approximations this test could be dispensed with if one has at his disposal a furnace with an electrode of this material, and the necessary facilities for varying the current and measuring the voltage, current and temperatures. If one of two accurately compared pyrometers is placed just beyond the hot end, and the other inserted in the electrode as near as possible to the hot end, and if voltage connections be made with the hot and cold ends (being careful to avoid thermo e. m. f's); then if the current be gradually increased until the two pyrometers read alike, the constants could be calculated for redesigning the electrode more correctly or for a different current. In that case it should not be forgotten that the length and the watts are only half of those in the formulas given in the present paper.

It was apparently not questioned that this method of determining the true average values of the conductivities to be used in the simple formulas for calculating electrodes given in my other paper, gives correct results in practice no matter what the law of variation with temperature is, or how great this variation may be. Hence it seems that the academic criticism of the other paper concerning a minute temperature correction of the formulas, does not apply when the results from this test are used with those formulas. Perhaps some academician will determine this with great precision for those who seem to want academic accuracy; meanwhile the present method will, I hope, give the practical engineer at least something better than what he has had heretofore.

A paper presented at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909; President L. H. Baekeland in the Chair.

FURNACE ELECTRODE LOSSES.

By C. A. Hansen.

THE CALCULATION OF ELECTRODE LOSSES.

In this paper some material appears which also appears in nearly the same form in Mr. Hering's papers. As I do not wish to be considered a plagiarist, I wish to explain that my paper is based upon formulæ courteously submitted by Dr. E. F. Roeber, who also wrote me that he acknowledged Mr. Hering's priority in the matter and asked me to credit Mr. Hering with the equations.

As Mr. Hering points out in his article on the above subject,[1] it is obviously "cheaper, quicker and simpler" to calculate the dimensions of a minimum loss electrode than it is to arrive at these dimensions by experimental means. That the results obtained by calculation, based, as they are, upon "constants" which vary between wide limits and concerning whose variation very little is known, lead to any feeling of security on the part of the designer, is not by any means so obvious. Neither do the calculated results lead to the evaluation of any of the "constants" concerned.

At one of the earlier meetings of the American Electrochemical Society, Mr. FitzGerald was asked by some one, Mr. Hering or Dr. Richards, I believe, whether he had calculated the heat losses for a certain furnace. Mr. FitzGerald has had a wide and varied experience with electric furnaces, yet he replied that he had *tried* to calculate his losses, but "had given it up as a bad job."

This is without doubt a regrettable state of affairs, yet one which I feel certain that most of those who are daily concerned with furnace work will concede exists.

I shall, however, try to analyze the electrode losses in one of our furnaces, concerning which I have already published con-

[1] Electrochem. and Met. Ind., **7**, 400.

siderable data, and to supply such data concerning "constants" as is derivable from the work done here.

This work, by the way, is not intended to be, and I had not

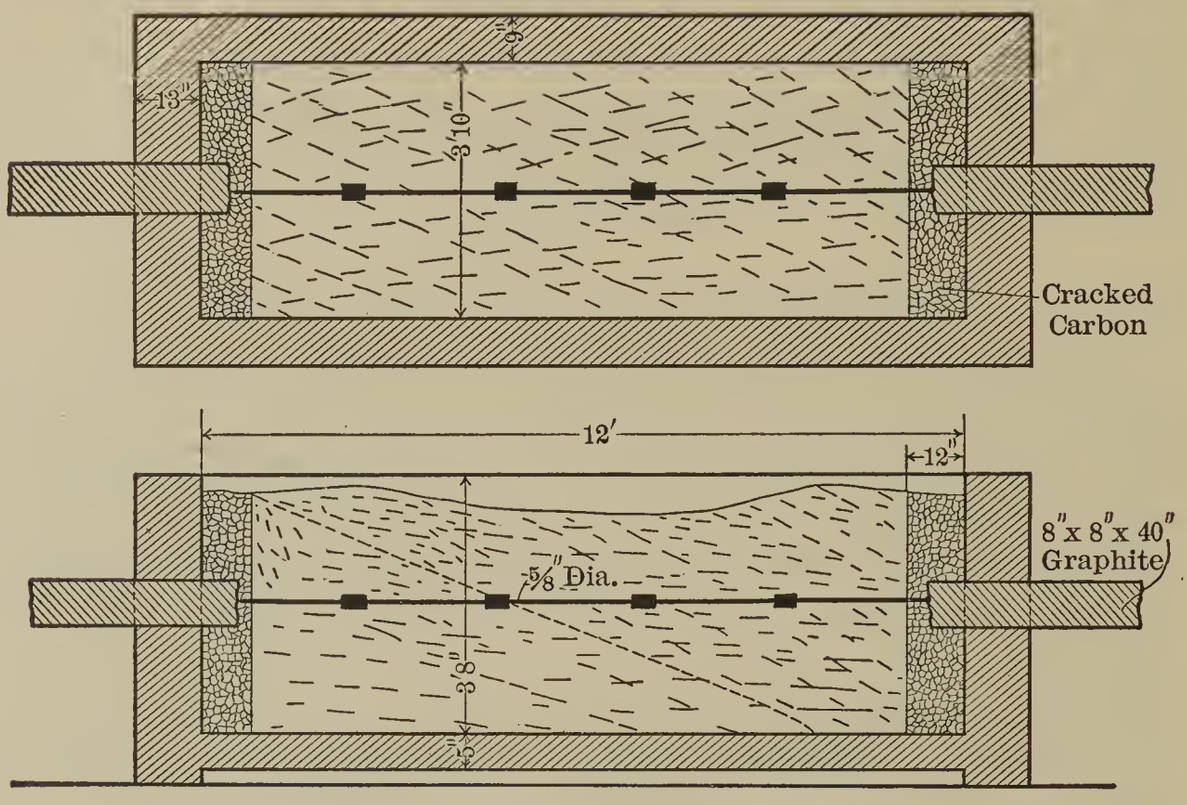


FIG. 1

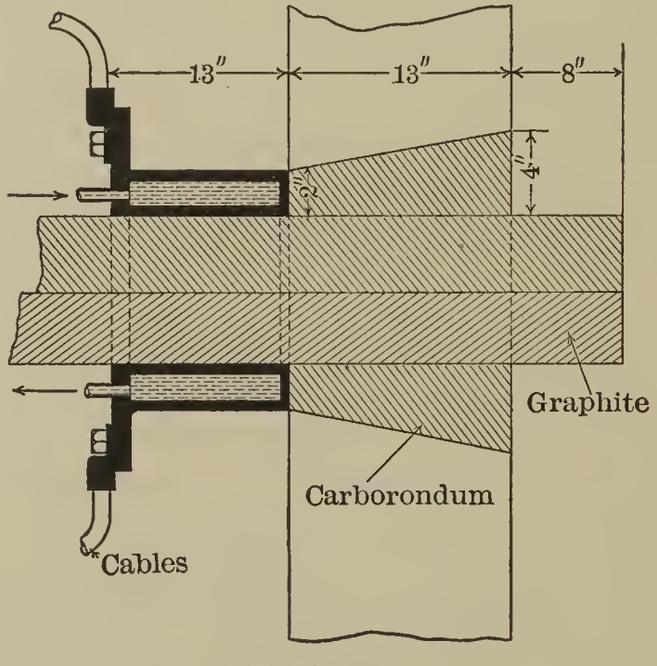


FIG. 2

thought of designating it as, an "exhaustive experimental research." With but two exceptions, so far at least, all of the experiments described have had a primary object in view other than the determination of electrode losses. The loss determina-

tions were entirely secondary, but as they seemed interesting, I have published them. In every case the attempt has been made to subordinate the discussion of results to the actual experimental data, and so far as possible, all of the existing conditions which concern the loss data are included in the article. It was utterly impossible to foretell when another relevant experiment could be carried out, so the results have been published piecemeal, with the intention of summing up when sufficient material had been gathered.

The furnace dealt with is shown in Fig. 1, the electrode design in Fig. 2.

The furnace has been so charged at different times that it would finish its run at 10,000, 15,000 and 20,000 amperes respectively, being kept at these current values for several consecutive hours in each case. The losses as determined in the cooling water under these conditions remain fairly constant, showing that in all probability an equilibrium has been established within the electrode. The temperature at the hot electrode end has been measured as described in a previous article[2] and for each case was found to be about 3200° C. The cold end of the electrode reached a temperature of approximately 200° C. The temperature difference was therefore 3000° C.

Let us analyze the losses to see what can affect them and how far these losses are determinable from published data.

First. Joulean heat, or

$$C^2R = \frac{C^2\rho l}{S}$$

(*a*) Current can be measured accurately enough for all practical purposes.

(*b*) Regarding the resistance of Acheson Graphite, the only figure I can find is 0.00032 ohm per inch cube, given as the cold resistance by the Acheson Graphite Company. No mention of a temperature coefficient is made.

(*c*) What length of active electrode shall we use? The resistance of the terminal head is so much lower than that of the electrode within it that perhaps all of the current passes into the electrode at the extreme tip next the furnace. The electrode end within the furnace may

[2] Trans. Am. Electrochem. Soc., **15**, 279.

disseminate the current in the charge all along its length. Obviously, all of the electrode length is not the resistor for the whole current carried.

(*d*) As regards section, that is easier, for in all probability the carborundum packing, although it has been known to carry current (Carborundum furnace), does not carry any large proportion of the current. "Skin effect" does not enter at 40 cycles, since, as nearly as I can estimate

$$\frac{\text{Rac (40 cy.)}}{\text{Rdc}} = 1 + 0.000025 - 0.0000000005, \text{ etc.}$$

This is due to the high specific resistance of the material.

To sum up, of the four terms necessarily involved:

C—is known.

S—is known closely enough.

l—May vary 50 per cent. easily, depending upon assumptions made.

ρ—Value for cold material known, but no part of the electrode is cold.

Second. Heat conductivity.

$$H = K \frac{(T_2 - T_1)\ S}{l}$$

(*a*) K is known for Ceylon graphite and a few other varieties, but not, so far as I know, for Acheson Graphite.

(*b*) $(T_2 - T_1)$ would be assumed anyway, if the calculation were to be made. In this particular case we know closely enough what it is.

(*c*) S and l are again open to the same speculation, as in the case of the C^2R loss.

To sum up:

K—is not known.

$(T_2 - T_1)$ is assumed—in this case known.

S and l as in the case of the C^2R loss.

Third. Contact resistance is neglected altogether in the formulæ so far published.

The formula used by Mr. Hering—

$$\frac{C^2R}{2} + H = \text{Loss}$$

tells us that one-half of the Joulean heat developed in the electrode, plus that heat which would be conducted were the electrode merely subjected to the same temperature difference across its extremities, goes into the cooling water or into the radiator, depending, of course, upon whether or not the electrode is water-cooled.

This is approximately true under certain restricted conditions. Under certain other conditions much of this heat quantity $(\frac{C^2R}{2} + H)$ does not get so far as the cooling water. This is not, however, exactly an efficiency gain. When the current does not exceed 10,000 amperes in the electrode shown in Fig. 2, the end wall remains intact. If we assume in that case that the electrode is maintained at an average temperature of 1200° C., is surrounded by a silica brick wall 10 inches thick, the outside of which is kept at room temperature, the loss of heat through the wall would only be 3.5 kw., and half of this only would, strictly speaking, be an electrode loss. This is, of course, only a very rough approximation.

When the current is increased to 20,000 amperes in the same electrode, a volume of silica brick, weighing some 100-125 pounds, disappears during each run. In fact, it is almost possible to correctly guess the ammeter reading by watching the furnace fumes. Now to vaporize 100-125 pounds of silica requires energy which would otherwise appear in the cooling water, but the silica passes up through the furnace charge and must naturally lose some of that energy as it passes through the cold top layer.

One kw. hour per pound of SiO_2 vaporized may be hazarded as a guess at the energy quantity thus removed, one-half of which should properly be added to the cooling water readings.

With this pessimistic analysis before me, I should certainly not feel any degree of assurance that I could come within hailing distance of an electrode loss.

Some of the missing data I can assign approximate values, not, however, arrived at by calculation other than such as is based on a reasonable amount of experimentally determined fact.

Resistance.

In May, before this Society, I showed a table of resistances of Acheson graphite electrodes. It was intended that this table should be published, but because of an unintentional delay on my part it did not appear in the Transactions. This table is now given here:

Resistance of Acheson Graphite Electrodes.

Size of Section	Sample Taken	Number of Samples	Ohms per inch Cube
⅛ inch diameter	Lengthwise	3	0.000391 -0.000396
¼ inch "	"	3	0.000343 -0.000379
⅜ inch "	"	3	0.000329 -0.000353
½ inch "	"	3	0.000268 -0.000290
1 inch "	"	2	0.000885 -0.000898
2 inch "	"	2	0.000363 -0.000367
3 inch "	"	2	0.000405 -0.000433
3 inch "	Crosswise	2	0.001025 -0.001030
4 inch x 4 inch	Lengthwise	2	0.000378 -0.000399
4 inch x 4 inch	Crosswise	2	0.000802 -0.000802
6 inch diameter	Lengthwise	2	0.000329 -0.000334
6 inch "	Crosswise	2	0.000655 -0.000662

The figures given are the extremes of 12 measurements made on each sample at about 25° C. The samples were cut from material taken from our own stock in April, 1909.

As regards the temperature coefficient, measurements were made on a ⅛-inch diameter rod in vacuo, with the following results, which I believe represent the true resistance at a temperature not over 50° C. from that given:

Temperature, ° C.	Resistance, %.
25	100
400	94.0
800	81.5
1200	66.0
1600	65.0
2000	68.0
2200	69.0

It may also be well to place on record some measurements on National carbon electrodes taken from our own stock at various times within the last three years.

RESISTANCE OF NATIONAL CARBON CO. ELECTRODES.

Size of Section	Sample Taken	Number of Samples	Ohms per inch Cube
¼ inch diameter	Lengthwise	5	0.00180 -0.00113
½ inch "	"	5	0.00113 -0.00147
⅝ inch "	"	19	0.00132 -0.00193
4 inch x 4 inch	"	1	0.00180
4 inch x 4 inch	Crosswise	2	0.00225 -0.00252
6 inch x 6 inch	Lengthwise	1	0.00337
6 inch x 6 inch	Crosswise	2	0.00350 -0.00370
8 inch x 8 inch	Lengthwise	2	0.00234 -0.00280
18 inch x 18 inch	"	3	0.0056 -0.0100

In justice to the National Carbon Company, it must be said that the 18" x 18" x 72" carbons, last referred to, were made over three years ago, and at the time, I believe, they were the largest ever made in this country. I understand that large electrodes recently made are very much better, and in that case I know the resistance will be found to be very much lower than the values given.

I have also been guilty of speculating as to the probable effect of temperature on the resistance of carbon, with curious results.

Samples of ⅝" diameter carbon rods obtained from the National Carbon Company were fired in groups of three at various temperatures in an Arsem vacuum furnace. One batch of four samples was fired in the electric tube furnace, mentioned in a previous paper[3] to 3500° C.

Average cold resistance values, etc., for the fired samples are given:

Temperature °C.	Loss in Weight Per cent.	Linear Shrinkage Per cent.	Cold Resistance of Fired Samples Per cent. Original Cold Resistance
1200	0.15	0.53	91.6
1600	0.56	0.40	87.0
2000	1.29	1.19	77.6
2400	1.64	2.38	65.9
2800	1.93	0.52	50.9
3500	2.43	0.117	22.4

[3] Trans. Am. Electrochem. Soc., **15**, 290.

The resistance of the 3500° samples, *i. e.*, 0.000307 ohms per inch cube, is practically the same as that given for Acheson graphite.

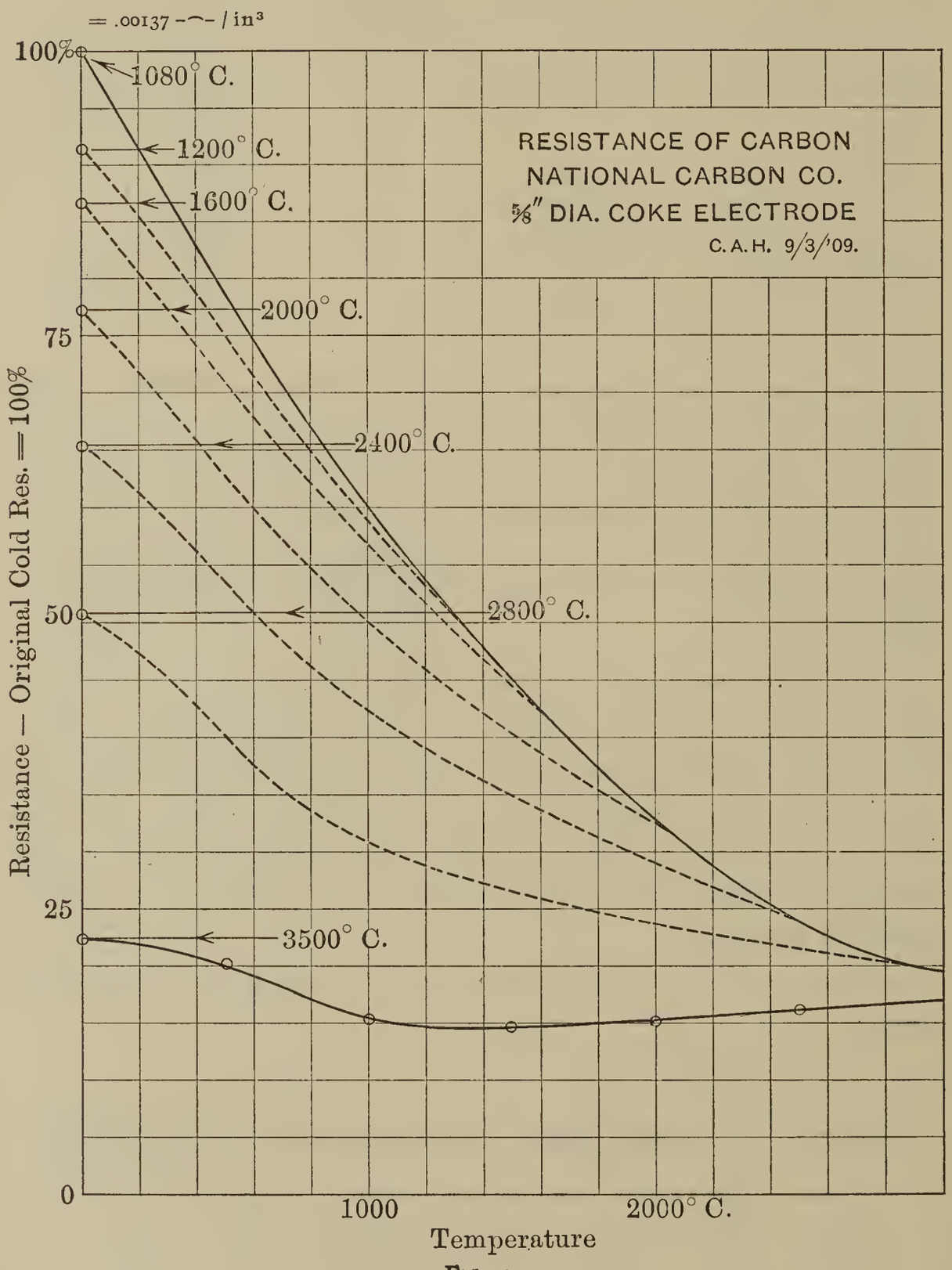


FIG. 3.

Fig. 3 shows where the speculation as to the temperature resistance diagram for carbon comes in. If we attempt to measure the temperature coefficient of resistance and, in the

course of our measurements go to a temperature higher than that at which the carbon was originally fired, the curve will not return upon itself on cooling down. For instance, if we go to 1200° C. and cool off, the curve will return not to 100 per cent., but to 91.6 per cent.; if cooled down from 1600° it will return to 87 per cent., etc., until finally, if fired to 3500° C., it will return upon the curve for graphite, since the amorphous carbon is no longer amorphous carbon, but graphite perfectly comparable with the Acheson material in every respect. There is no reason to suppose that, when we exceed the maximum temperature to which the carbon has previously been fired, there is a break in the resistance curve. The curve for any carbon will be a smooth one, hence the dotted curves are all drawn tangent to the parent curve. For all of the commercial carbons, the temperature coefficient of which I have measured, the 1200° hot resistance is around 60 per cent. of the cold resistance. Upon the assumption that the 1200° resistance is about 60 per cent. of the cold resistance for National Carbon Company electrode material fired to various temperatures and upon the assumption that all of the branch curves are tangent to a common curve, the extreme ends of which are located, the curves shown are a reasonable derivation.

If, now, we assume a straight line gradient in our electrode and a temperature difference between hot and cold ends, as in the case under consideration, of 3000° C., the effective resistance of our graphite electrode will be the true average resistance of graphite as determined by averaging a lot of resistance ordinates equally spaced along the temperature axis of the temperature-resistance diagram for graphite. So, for graphite, this is fairly simple when we have the "constants." For $T_2 - T_1 = 3200° - 200°$ C., the average is about 78 per cent. of the cold resistance.

With carbon, on the other hand, if the electrode has been alternately heated and cooled several times, different points in its length being heated to different temperatures, the working resistance of the electrode may be anything at all. Any point within the triangular area of Fig. 3 will be sure to fit out some point in the electrode with its proper "constant." Let us assume, however, that, as for graphite, the average ordinate along the

parent curve holds. For our furnace and carbon electrodes, the effective resistance is then 40 per cent. of the original cold resistance, or, for 4″ x 4″ x 40″ electrodes, 0.00072 ohms per inch cube.

Electrode resistance was actually measured under the operating conditions. The results will be taken up later.

Effective Length of Electrode and Contact Resistance.

These are treated together, for, as we shall see, they are to a certain extent interdependent.

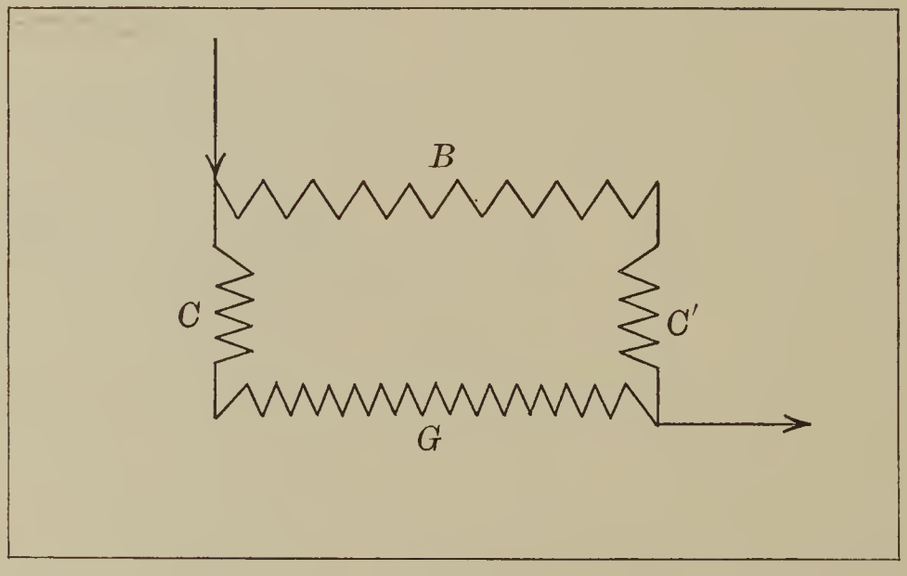


Fig. 4.

The length of our electrode may be divided into three portions and the portions treated separately. These portions are:

1. The length within the furnace itself.
2. The length embedded in the furnace wall.
3. The length within the brass terminal.

1. Inspection of the experimentally determined temperature gradient for a carbon electrode similar mechanically to the one under consideration[4] leads to the conclusion that practically all of the current is carried to the extreme electrode tip, making this entire length an effective length.

2. It is safe to assume that practically all of the current is carried by the electrode portion embedded in the furnace wall, making this entire length an effective length.

[4] Trans. Am. Electrochem., **15**, 292.

3. With the length within the brass terminal, which is over 38 per cent. of our entire length, contact resistance enters to affect the effective length of conductor.

Let us consider the terminal and electrode multiple conductors from cable contact to the terminal tip next the furnace wall, and that the contact between the two is equally divided between the two ends of the terminal. (See Fig. 4.)

B = resistance of brass terminal.
G = resistance of graphite electrode.
C = C^1 = contact resistance.

The ratio of the current carried *via* BC^1 to that carried *via* CG becomes

$$\frac{C + G}{B + C}$$

G, taking 0.000350 ohms per inch cube = 0.000071 ohm.

B, taking brass at 0.0000032 ohm per inch cube = 5 times resistance of copper = 0.0000013 ohm.

If we neglect contact resistance, we find that 98.2 per cent. of the current goes *via* BC^1, as against 1.2 per cent. *via* CG. In other words, the current will be crowded into the electrode at the terminal end next the furnace wall, making very little of the length within the terminal an effective length. If the contact resistance is great as compared with G and B, then the current will be evenly distributed throughout the contact area, making one-half the length in the terminal effective conductor for the whole current, but in that case contact resistance must inevitably be a factor for consideration in calculating the electrode losses.

Some time ago I built a furnace with the resistor made up of carbon motor brushes, each 2″ x 2″ x 7/16″, with a resistivity in inch ohm units of 0.00120. These brushes were piled flatwise in a vertical column and were in good mechanical contact with one another. Pressure was applied to the column by means of a screw, but the pressure was not measured. I should judge it to be in excess of 200 pounds or over 50 pounds per square inch of contact.

The cold column had a contact resistance over 100 times the resistance of the brushes themselves, figured as a solid carbon

column. At 1800° the contact resistance was at least 20 times the resistance of the carbon.

This indicated that it would be worth while to look into the contact resistance question a little more closely.

Accordingly, an attempt was made to actually measure the contact resistance of the furnace terminals under operating conditions. It would perhaps have been simpler to measure the contact resistance of a column of plates, alternating graphite with

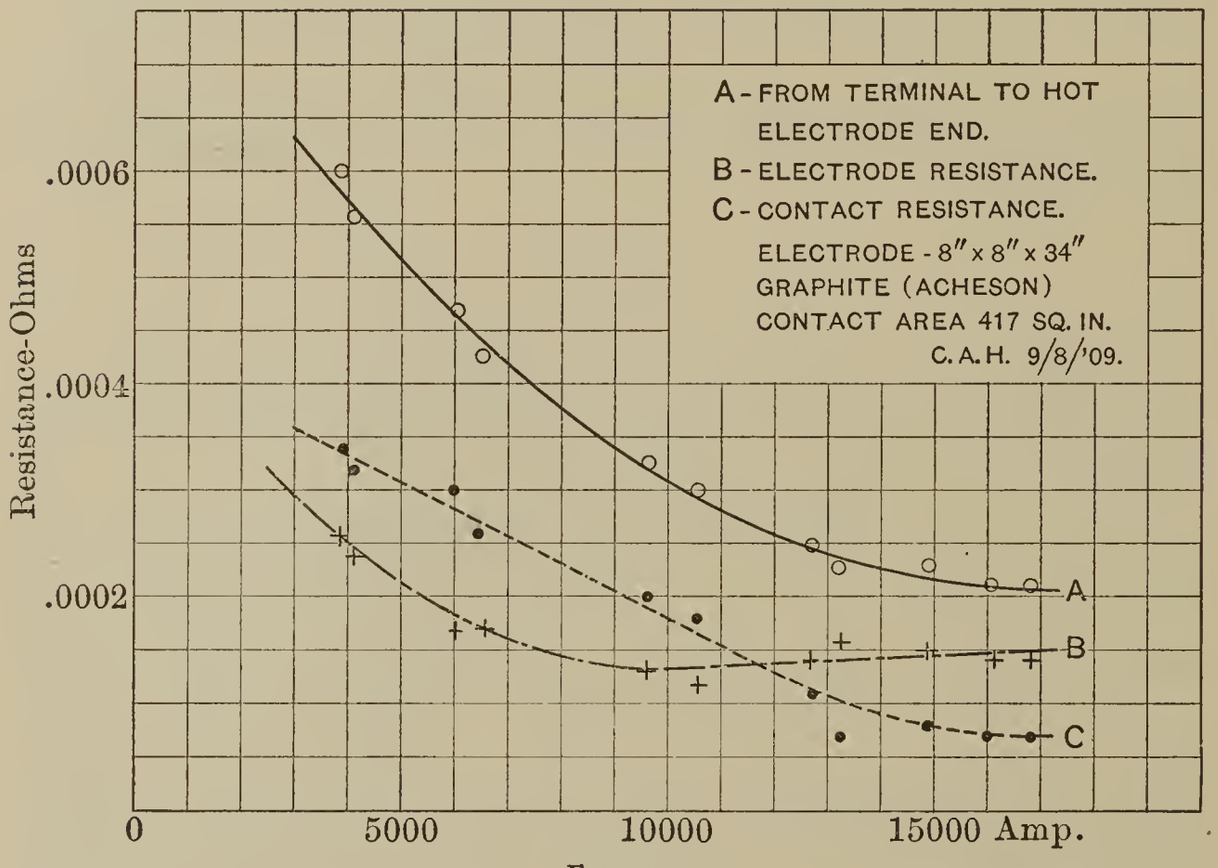


FIG. 5.

brass, and it was intended to do this in case the projected experiment did not yield results.

It is no easy or extremely accurate matter to determine the resistance of a conductor carrying 15,000 or 20,000 amperes alternating current by means of a voltmeter, and the results obtained are intended merely to give the approximate magnitude of the loss.

A 1¼-inch diameter graphite rod was threaded vertically into the tip of electrode within the furnace, and this rod projected some four inches above the charge. A terminal was threaded to the end of the graphite rod, another into the cold end of the

electrode, and a third connection was made to the brass terminal. The voltmeter leads were twisted together and brought out to the voltmeter, which, of course, was protected as much as possible from stray fields. The only loop in the voltmeter circuit was the rectangle bounded by electrode, graphite rod, voltmeter lead and furnace wall, 34″ x 26″ in dimension.

The results obtained were as follows; E being the potential drop: (Graphite electrodes as shown in Fig. 2.)

RUN OF SEPTEMBER 8, 1909.

Time	Current	Terminal to Hot Electrode End			Tip to Tip of Electrode			Contact			Water KW.
		E	KW.	R. Ohms	E	KW.	R. Ohms	E	KW.	R. Ohms	
8:30	Start										
9:30	3840	2.3	8.80	0.00060	1.0	3.84	0.00026	1.3	4.96	0.00034	2.4
10:00	4080	2.3	9.40	0.00056	1.0	4.08	0.00024	1.3	5.32	0.00032	2.6
10:30	6000	2.8	16.80	0.00047	1.0	6.00	0.00017	1.8	10.80	0.00030	4.5
11:00	6480	2.8	18.15	0.00043	1.1	7.13	0.00017	1.7	11.02	0.00026	5.2
11:30	9600	3.2	30.70	0.00033	1.2	11.51	0.00013	2.0	19.19	0.00020	12.0
12:00	10500	3.2	33.60	0.00030	1.3	13.65	0.00012	1.9	19.95	0.00018	14.0
12:40	12700	3.2	40.70	0.00025	1.8	22.85	0.00014	1.4	17.85	0.00011	20.5
1:10	13200	3.1	40.90	0.00023	2.0	26.40	0.00016	1.1	14.50	0.00007	21.0
1:45	14900	3.4	50.70	0.00023	2.2	32.80	0.00015	1.2	17.90	0.00008	29.9
4:15	16080	3.4	54.7	0.00021	2.2	35.40	0.00014	1.2	19.30	0.00007	37.5
4:45	16800	3.6	62.0	0.00021	2.3	38.60	0.00014	1.3	21.80	0.00007	36.8
5:10	17280	Cut off power, 3272 Kw. hrs. in furnace.									

Actual water record was taken to correspond only with the three last readings. The other water losses are taken from previous runs; they all check to within a few per cent. when compared on the basis of current if the power curves are at all comparable, and, as a rule, they are comparable. The terminal was in excellent shape mechanically—good contacts—drawn up as tightly as 4 standard ¾-inch bolts would safely stand without stripping threads.

The resistance data is plotted in Fig. 5.

Contact resistance is certainly a factor worth considering. To get back, then, to Fig. 4:

$$B + C = 0.0000013 + (2 \times 0.00007) = 0.0001413 \text{ ohms.}$$
$$C + G = (2 \times 0.00007) + 0.000071 = 0.000211 \text{ ohms.}$$

Current carried via BC = 60 per cent. as against 40 per cent. via CG. The resistance of the multiple circuit is 0.0000845 ohms,

which leaves only 0.0000145 ohms for the graphite electrode. In other words, only 20.4 per cent. of this portion of our electrode is effective conductor for all of the current. It should also be added that decreasing the contact resistance or increasing the resistance of the electrode itself diminishes this effective length percentage. This conclusion holds for heat resistance as well, but we cannot at present define the effective length of heat conductor so closely, because of our lack of knowledge concerning the heat conductivity constants.

For the purpose of this paper, however, I think we are justified in making the 20 per cent. value above developed a general value.

Also contact resistance, which has so far been neglected in all formulæ for the calculations of electrode losses, must somehow be brought into those formulæ. Its value is dependent upon current density at least, and as well upon temperature of contact, nature of contact, whether oxidized or not, etc., but for our own purpose, since all but current density is fairly constant, the values per square inch for it may be obtained by multiplying the resistances given in curve form (Fig. 5) by 417 — the contact area in square inches.

It is also curious to compare the contact losses here measured with some which I proposed last May as perhaps giving the magnitude of this loss.

"Contact Resistance Between Waterjacket and Electrode.

"Based on brush to commutator resistance at 2 pounds per square inch pressure, equivalent to 832 pounds distributed pressure on an 8-inch by 8-inch electrode in a 13-inch waterjacket.

1. 10,000 amps. in terminal = 24 amps. per sq. in. contact.
   ([5]) r = 0.036 R = 0.0000865 C^2R = 8.65 kw.

2. 15,000 amps. in terminal = 36 amps. per sq. in. contact.
   r = 0.026 R = 0.0000625 C^2R = 14.05 kw.

3. 20,000 amps. in terminal = 48 amps. per sq. in. contact.
   r = 0.023 R = 0.0000553 C^2R = 22.12 kw."

([5]) r = resistance per square inch of contact.

Comparing these results with the data determined, we have:

Current	R	C^2R	R	C^2R	
10,000	0.0000865	8.65	.00018	19.95	
15,000	0.0000625	14.05	.00007	19.30	
20,000	0.0000553	22.12	.00006	24.00	(exterpolated value)

Now that we can safely judge the effective length of conductor, we can also arrive at fair valuations of both electrical resistivity and heat conductivity, for I do not think it unreasonable that the same effective length holds for both.

Electrical Resistivity of Graphite.[6]

Electrode composed of 4 pieces 4″ x 4″ x 40″ bundled together.

Time	Current	Resistance	Resistivity (Ohms per inch Cube)
9:30	3840	0.00026	0.00070
10:00	4080	0.00024	0.00064
10:30	6000	0.00017	0.00045
11:00	6480	0.00017	0.00045
11:30	9600	0.00013	0.00035
12:00	10500	0.00012	0.00034
12:40	12700	0.00014	0.00037
1:10	13200	0.00016	0.00043
1:45	14900	0.00015	0.00041
4:15	16080	0.00014	0.00037
4:45	16800	0.00014	0.00037

The resistance of an average Acheson graphite electrode used with a temperature range, $T_2 - T_1 = 3200 - 200°$ C., by actual measurement is then 0.00037 ohm per inch cube.

By cold resistance and temperature-resistance diagram, the same electrode should have a resistivity of 0.00030 when the lengthwise resistance figure is taken.

Considering the opportunities for error in the determination of the resistance of the electrode as a whole, I prefer to adopt the lower value of resistivity, and to use the value 0.00032 ohms per inch cube as the mean working value between these extreme limits of temperature variation.

I wish here to say that Mr. Williamson's comment relative to the resistivity of graphite used in a former paper was replied to in the discussion of that paper. I used the average of cross-

[6] Same run detailed above.

wise and lengthwise resistivities, since it seemed some recognition of the high cross resistance should be made when short electrodes were used. In the light of these developments, I do not now believe that it is necessary to use anything but the lengthwise resistivity. It is interesting to note, however, that it is always possible to determine, by resistance measurements, whether a carbon was extruded from a die or merely formed under pressure, and that it is also possible to determine the axis along which the material was extruded, or "squirted."

Heat Conductivity.

From the experimental data obtained by measuring the cooling water losses of carbon and graphite electrodes not carrying current, as previously described (p. 286, Vol. XV), it is also possible to assign conductivity figures to both carbon and graphite for extreme differences in temperature.

Graphite "idler":

$l = 12.75''$ $\qquad T_2 = 2830°$ C.

$S = 16.0$ $\qquad T_1 = 30°$ C.

Loss = 3,580 watts.

K for graphite = 1.65 watt seconds / sec. / ° C. / for 1 inch length and 1 square inch section.

= 0.162 (c. g. s. units.)

Carbon "idler":

$l = 12.75''$ $\qquad T_2 = 3500°$ C.

$S = 16.0$ $\qquad T_1 = 30°$ C.

Loss = 445 watts.

K for carbon = 0.165 watt seconds / sec. / ° C. / for 1 inch length and 1 square inch section.

= 0.0162 (c. g. s. units.)

In connection with my experiments comparing electrodes of similar sizes but of different materials, I wish to reply to Mr. Hering's criticism of this procedure:[7]

"Comparing different materials experimentally by means of electrodes of the same size is equally incorrect and misleading; it also leads one away from the goal, rather than toward it."

I fail to see how the comparison would be improved by resort-

[7] Electrochem. and Met. Ind., **7**, 400

ing to electrodes of different sizes. Grant that we did not have enough of an idea of "constants" involved to calculate a loss directly. Would we then gain by gratuitously making the conditions such that no single factor would be the same for the two materials to be compared? In either case, whether the electrodes are of the same size or of different sizes, a set of conditions is experimentally determined. Without resorting to doubtful "constants" and doubtful dimensions, a comparison of some kind may be made if the electrodes are of the same size, whereas without resorting to "constants," of which even the approximate magnitude was unknown, no comparison whatever is possible, if the electrodes are of unlike sizes. Again, in these experiments both electrodes—carbon and graphite—were run at current densities, which cannot be practically maintained even in the graphite electrodes, densities which were then extraordinarily high for carbon. The attempt was made to cover the entire practicable range of both electrodes, and comparisons at different current densities for the two electrodes can be made with greater security by interpolating the data obtained on like sizes than would be possible in the other case.

All this aside, we have now the most important data required for calculating losses.

1. Length: The entire length of electrode from *center of waterjacket* to extreme hot end.

2. (*a*) Resistivity of graphite under conditions applicable to a temperature difference of 3000° C. This is .00032 ohm per inch cube = .000812 ohm per cm. cube.

(*b*) Resistivity of carbon under same conditions may be taken as .00072 ohm per inch cube, or .00183 ohm per cm. cube.

Ratio of carbon resistance to graphite resistance under these conditions is 2.25.

3. (*a*) The heat conductivity of graphite 3200° C. — 200°. = 1.65 watt seconds per second per degree Centigrade for 1 inch length and 1 square inch section = 0.162 c. g. s. units.

(*b*) The heat conductivity of carbon under the same conditions is 0.165 watt seconds per second per degree Centigrade for 1 inch length and 1 square inch section = 0.0162 c. g. s. units.

4. Contact resistance may be taken as 0.075, 0.029 and 0.025 ohm per square inch for brass to graphite at current densities through the contact area of 24, 36 and 48 amperes per square inch respectively.

Calculation of Losses for Titanium Carbide Furnace Electrodes.

Material, graphite.
$T_2 - T_1 = 3000°$ C.
Section = 64 square inches.
Length = 23.6 inches.
Current = 10,000, 15,000, 20,000 amperes.

Heat loss is the same in each case.

$$H = K\frac{(T_2 - T_1)S}{l} = \frac{1.3 \times 3000 \times 64}{23.6} = 10.55 \text{ kw.}$$

$$C^2R \text{ loss} = \frac{C^2 \rho l}{S} = C^2 \times \frac{.00032 \times 23.6}{64} = C^2 \times .00011.$$

C = 10,000	C^2R = 11.8 kw. ,	$C^2R/2$ = 5.9 kw.
C = 15,000	C^2R = 26.6 kw. ,	$C^2R/2$ = 13.3 kw.
C = 20,000	C^2R = 47.2 kw. ,	$C^2R/2$ = 23.6 kw.

Total loss = H + $C^2R/2$ (no account taken of contact resistance).

According to this formula, then, the losses for 10,000, 15,000 and 20,000 amperes will be 16.45, 23.85, and 34.15 kw., respectively.

The contact resistance losses are 19.95, 19.30 and 24.00 kw., respectively for 10,000, 15,000 and 20,000 amperes.

If we add all of this to the losses previously calculated, we get respectively 36.40, 43.15 and 58.15 kw.

If we add half of it only, we get respectively 26.42, 33.50 and 46.15 kw.

The corresponding losses actually measured by cooling water methods are 22.00, 28.00 and 36.00 kw.

The losses as calculated from the curve sheets given in a previous paper[8] are 21.2—34.4, and, if we for once exterpolate the curve sheets given, 42 kw., respectively.

[8] Electrochem. and Met. Ind., **7**, 358.

Putting these results together in table form, we get:

Current	A	B	C	D	E
10,000	16.45	36.40	26.42	22.00	21.2
15,000	23.85	43.15	33.50	28.00	34.4
20,000	34.15	58.15	46.15	36.00	42.00

$$\text{A Loss} = \frac{C^2 R}{2} + H$$

$$\text{B Loss} = \frac{C^2 R}{2} + H + \text{Contact } C^2R$$

$$\text{C Loss} = \frac{C^2 R}{2} + H + \frac{\text{Contact } C^2R}{2}$$

D Loss = Measured in cooling water.

E Loss = Curves based on measured losses for 4 in. x 4 in. graphite electrodes.

The discrepancy between D and E at 15,000 and 20,000 amperes is, I believe, in part, a measure of the losses from electrode to furnace wall. The major part of the wall loss is, however, no doubt, due to volatilization of silica, which makes the actually measured losses low.

As a conclusion, then, I think that either column C or E (*i. e.*, either the losses calculated from loss =

$$\frac{\Sigma\, C^2R}{2} + H,$$

or the losses as calculated from the curve sheets given)[9] more nearly approximate the true losses than either the losses actually measured in cooling water *at high current densities*, or those calculated from heretofore published formulæ.

Fortunately, we can considerably improve upon our contacts by increasing the number of electrodes which make up a given section, and then making use of the increased surface afforded by these separate electrodes, as, for instance, by providing individual threaded caps.

Since in this case the contact area is not necessarily a function of the dimensions of the entire composite electrode, the equation $\frac{C^2R}{2} + H = \text{Loss}$, where C^2R refers only to the actual electrode resistance, should be of value in calculations for the minimum-loss-electrode dimensions.

[9] Electrochem. and Met. Ind., 7, 358.

At one point, however, in Mr. Hering's article, I find that current density is an entirely impossible quantity upon which to base electrode design.

Mr. Hering shows that the equation for electrode loss does not contain either length or section separately in any of its terms.

$$l \text{ and } S \text{ always appear as a ratio } \frac{l}{S} \text{ or } \frac{S}{l},$$

and solving the equation for minimum loss condition merely gives us the most economical ratio l/S. In that case, it becomes exceedingly difficult to determine what would be better to base dimensions upon than current density—certainly not either length or cost.

I should prefer, in connection with my own work, to take a current density of 150 amperes per square inch for graphite, and 50 amperes per square inch for carbon electrodes, since I know from experience that when I exceed these densities for any considerable period, my end wall is badly corroded. On this basis and solving Mr. Hering's equation for dimensions for a 20,000-ampere electrode:

$$\frac{l}{S} \text{ (minimum)} = \frac{1}{C}\sqrt{2\,(T_2 - T_1)\,\frac{k}{\rho}}$$

$$\frac{l}{S} = \frac{1}{20000}\sqrt{2 \times 3000 \times \frac{1.65}{.00032}}$$

$$= \frac{5570}{20000} = 0.278 \text{ (inch units used)}$$

At a current density of 150 amperes per square inch, the graphite electrode would have a section of 133 square inches and an effective length of 37.0 inches.

Calculating the dimensions of a carbon electrode to do the same work, I found that with the "constants" here given l/S should be 0.0585. At 50 amperes per square inch, this means an electrode 20″ x 20″ in section and 23.4 inches effective length.

The minimum loss for the graphite electrode would be 34.4 kw. if we can assume that contact loss can be reduced to zero. The minimum for the carbon electrode would be 16.9 kw., under the same assumptions.

The ratio of minimum losses is 2.04, and is in favor of carbon.

It is also evident that the equation

$$\frac{l}{S} = \frac{1}{C}\sqrt{2\ (T_2 - T_1)\ \frac{k}{\rho}}$$

may be converted into

$$l = \frac{1}{d}\sqrt{2\ (T_2 - T_1)\ \frac{k}{\rho}}$$

where d = current density.

If we assume that 50 amperes for carbon and 150 amperes for graphite are good current densities for general work, and that the "constant" K and ρ hold for most work, we can solve for length directly:

$$l = \frac{1}{150} \times \sqrt{2\ (T_2 - T_1)\frac{k}{\rho}} = 0.69\sqrt{(T_2 - T_1)}$$

for graphite and

$$l = \frac{1}{50} \times \sqrt{2\ (T_2 - T_1)\ \frac{k^1}{\rho^1}} = 0.43\sqrt{(T_2 - T_1)}$$

for carbon. l is expressed in inches, $(T_2 - T_1)$ in degrees Centigrade.

In furnaces of the Heroult type, operating conditions require current densities in the electrodes of 30 amperes, or less, per square inch, but in no other case that I recall would these equations have so little bearing in determining the electrode dimensions as in the present types of steel furnace. Protection for the exposed terminals and a maximum uninterrupted downward feed are the determining factors in this work, aside from the choice of an electrode section which is great, relative to the area of the bath surface.

Mr. Hering states that under certain circumstances the carbon electrode designed for minimum loss would have awkward dimensions. He has, however, admitted that under the same circumstances he might sacrifice efficiency, and change the dimensions of his carbon electrode to suit. This might be done and still not exceed the minimum losses of a graphite electrode.

As regards an electrode being so proportioned that it acts as a "heat valve," some point within it being at a higher temperature

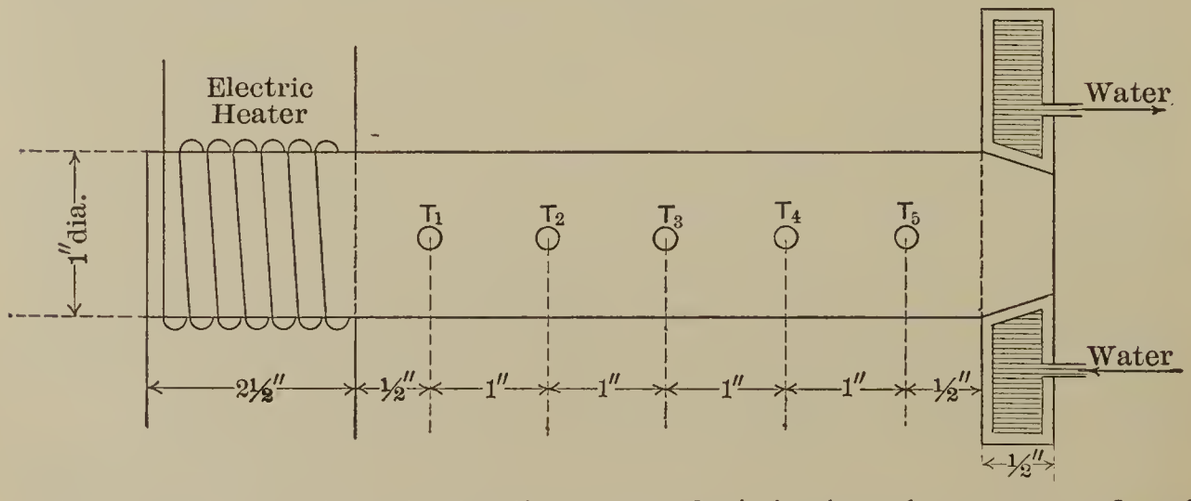
than the furnace interior, this is obviously an inefficient condition. That portion of the electrode between the "heat valve" and the furnace interior is merely a useless clog. In effect, he has gratuitously increased the value $(T_2 - T_1)$ and shortened the distance through which the increased temperature difference can drive heat into his cooling water or radiating surface, as the case may be.

The minimum electrode loss condition is, as Mr. Hering shows, that in which $\frac{C^2R}{2} = H$; and if this condition is reached as it is, or is supposed to be at the proper value of $\frac{1}{S}$, then the exact tip of the electrode is at exactly the temperature of the furnace interior. In this case $T_2 - T_1$ is a minimum and 1 a maximum. H is, therefore, a minimum, and since $H = C^2R$, the total loss is a minimum.

Research Laboratory,
General Electric Co., Schenectady, N. Y.
September 10, 1909.

APPENDIX.

Between the writing and the presentation of my paper, I have made a few heat conductivity tests, which, as they have a bearing here, properly belong with this paper.



A graphite rod, 1 inch diameter, 8½ inches long, was fitted

with a heating coil at one end, a water cooling head at the other and intermediate and evenly spaced were placed three thermo-couples and two mercury (370° C.) thermometers. The whole apparatus was packed in freshly burned powdered lime. Obviously all of the heat measured in the cooling water passed through the rod, and obviously no more heat passed through the rod than was generated in the coil. Granting the accuracy of the temperature determinations, then we can assign maximum and minimum values to the heat conductivity coefficient.

The results obtained were:

Heat Conductivity of Acheson Graphite.

In the Temperatnre Limits C°	At Mean Temperature	Max. K.	Min. K.	Mean K.
90- 110	100	5.90	4.76	5.33
180- 220	200	4.68	3.64	4.16
260- 340	300	4.08	3.24	3.66
350- 450	400	3.76	2.80	3.28
440- 560	500	3.52	2.60	3.06
500- 700	600	3.34	2.40	2.87
2800-3200	3000	From Electrode Experiments		0.02

Heat Conductivity of National Carbon Co. Carbon.

(Apparently made of petroleum coke.)

In the Temperature Limits	At Mean Temperature	Max. K.	Min. K.	Mean K.
37-163	100	0.298	0.030	0.164
105-225	165	0.293	0.071	0.182
105-225	165	0.293	0.071	0.182
160-325	242	0.188	0.049	0.118
160-325	242	0.188	0.049	0.118
170-330	250	0.293	0.046	0.170
200-340	270	0.306	0.061	0.185
240-523	382	0.217	0.031	0.124
263-543	403	0.217	0.043	0.130
283-597	440	0.115	0.048	0.132
	3000	From Electrode Experiments		0.02

K, in all cases = Watt seconds / second/ °C /sq. in. / inch/

The errors due to loss of heat along the rod were only nominal as compared with the heat passed through the rod in the graphite experiments, so that the actual value of conductivity coefficient is placed within fairly narrow limits. For carbon, the heat loss from the side of the rod was enormous compared with the heat that passed through, and the method cannot be said to be in any

way satisfactory. It is gratifying, however, to note that Mr. Fitz-Gerald fixed a minimum value for the ratio K for graphite/K for carbon, when he gave the figure 18.1 at low temperatures.

The graphite values are also well above the corresponding values of the metals. For instance:

K (Watt-secs / sec / °C / sq. in. / inch.)

Silver	11.6
Copper	11.0
Graphite (100°C.)	5.3
Zinc	3.2
Iron	1.8

I also wish to point out that in some ways these determinations place graphite in an anomalous position. I believe that it is usual for the heat conductivity to increase with temperature in those cases in which the electrical conductivity increases with temperature and *vice versa*. Here we have a case in which heat and electrical conductivities vary in opposite directions.

DISCUSSION.

Dr. E. F. Roeber (*Communicated*): To the statement of Mr. Hansen concerning the origin of and credit for the formulas used by him, I may add that I sent my own calculations (which I had made independently of Mr. Hering, and without knowing his formulas) to Mr. Hansen before I had received or seen the manuscript of Mr. Hering's article, which latter was published afterwards in the October issue of Electrochemical and Metallurgical Industry, and which was the first publication of Mr. Hering's formulas.

Mr. Carl Hering: I have prepared quite a long answer to Mr. Hansen's paper, but I feel that it is not of sufficient general interest to burden you with it at this meeting. I will therefore contribute it as a communicated discussion and merely call attention here to a few points, some of which he has alluded to in his present abstract, which dealt chiefly with new matter which was not in the paper.

He shows a curve which he says is mine (I have not had the opportunity to check it), and one which he says is his, and he

says they do not agree. My curves refer to carbon—I wish to emphasize that. His curves do not refer to carbon; they refer to something which was, but is no longer carbon. I would warn anyone from accepting Mr. Hansen's figures for carbon, as the constants for carbon, because they are not the constants for that material; they are the constants for carbon which has been more or less graphitized, presumably more rather than less, so that I am sure one would get erroneous results if one used Mr. Hansen's figures for carbon in calculations for furnaces where the temperature does not go up to the graphitizing point; such, for instance, as iron and other furnaces, where carbon is not graphitized as it is in Mr. Hansen's furnace. Mr. Hansen based these deductions on a furnace with an exceedingly high temperature; he has gotten certain results, but I do not think it would be right to apply these results to lower temperature furnaces. They would be very misleading.

His figures for carbon are very favorable for economy of power in electrodes, and those for graphite are less so. The graphite figures I do not quite understand, because in one part of the paper he gives one figure for graphite, and in the latter part he gives a rather more unfavorable figure, which leads to the impression that the latter was a poor quality of graphite, possibly "home-made" graphite, like that which he gets when he graphitizes carbon in the form of electrodes. However, the difference between his and my figures for graphite does not really amount to much; in fact, his figures tend rather to confirm roughly those that I used, which were deduced from his own interesting experiments. Some months ago he obtained a ratio of 1 to 8, and now he obtains a ratio of 1 to 10 for the relative heat conductivities of carbon and graphite. He does not explain why he gets that difference now. I understand the original experimental data was the same.

The experimental information which Mr. Hansen has published from time to time is very valuable, and I unhesitatingly acknowledge having benefitted from it, but I cannot agree with him in all of his deductions. I think his deductions are in part very misleading. I believe they have even misled him. In one of his published articles he explains that in one of his furnaces he burned out the walls frequently. If he had applied my formulas,

using his own constants, he would probably have found that he was getting the greatest temperature in his electrode within the furnace walls. When his electrode was being used with the largest currents, namely 15,000 to 20,000 amperes, he was heating his electrode to the highest temperature in the interior of the walls, and there is no wonder that he destroyed his furnace wall. If Mr. Hansen had used the formulas given in my paper, which he has criticised, he would probably not have repeatedly destroyed his furnace; he would then have made the electrodes either shorter or else larger in section.

In a paper published some time ago he concludes from these experiments, that electrodes should be made longer, while as a matter of fact it seems they were already too long in his own furnace, and if he had used the formulas given here he would have found that to be the case, so I think that Mr. Hansen's own deductions have misled him.

He still adheres to the old current density formula, although I think my paper has shown conclusively that that is an entirely wrong way to calculate electrodes. We used to do the same thing thirty years ago in calculating transmission lines, but abandoned it long ago; the same reasoning applies to electrodes to a great extent.

Mr. Hansen's diagram, Fig. 3, is very interesting, but it lacks entirely that shotgun effect which is usual with experimental curves; it is too regular. It is interesting to notice the very point which I raised, that for the lower temperatures, that is, the temperatures of most furnaces, the resistivity of carbon comes back to nearly the starting-point. Of course, if carbon is used far down this curve, one will get something that is not carbon at all, and it is therefore not right to call these constants the constants of carbon, as it is not carbon any more, but a poor quality of graphite. It is an unknown mixture of graphite and carbon, and hence it cannot be expected that the constants of that unknown mixture are reliable or uniform. If he obtains variable and unreliable constants from such a mixture, it is no wonder, because it is a mixture and not a specific material.

He calculates, with his own figures for carbon, that he ought to use a carbon electrode which is practically a cube, and therefore impracticable. He said near the end of his paper that this might

be changed somewhat, to sacrifice a little efficiency and get a longer electrode. I wish to ask Mr. Hansen how he proposes to change such an electrode to sacrifice efficiency and get a better length. If he says the electrode should be lengthened, let us see what will happen. We know the temperature at the end of the original cubical electrode will be about 3,000 degrees. If he lengthens it, he will get the 3,000 degrees (or even more) within the walls. Hence, lengthening the electrode will not help. Let us try to increase the cross-section. If we do so, we will undoubtedly get the high temperature within the wall again, and the furnace will burn out again.

I would therefore like to know what he expects to do to get a better shape of electrode with carbon, using his own figures. The only thing that I can see is to make the electrode very much larger in both length and section, so as to have the ratio of cross-section to length approximately what is required by theory, and then his electrode will probably not burn out. This means that his electrode may be nearly as large as the furnace itself. I am asking for information, when I ask him to tell us about that, for I am looking for information myself.

I might say, in this connection, that a graphite electrode for this furnace would be much smaller in section, which gives it a proportion which can be carried out in practice.

The rest I will answer in writing.

Mr. Carl Hering (*Communicated*): In addition to my remarks at the meeting, I desire to make the following comments:

Mr. Hansen's theoretical calculation of the contact resistance based on his Fig. 4, is not quite correct, as the surface resistance indicated by C and C^1 is distributed over the whole contact and is not concentrated at the two ends as shown, unless it is a fact that such a distributed resistance is the equivalent of two concentrated resistances; if so, it would be of interest if he had said so. The difference, however, is probably of no great importance, for at best any such theoretical or speculative calculations of a contact resistance must necessarily be crude.

In connection with that interesting curve, Fig. 3, he also gives the values for carbon for the lower temperatures, such as are used in the iron furnaces, for instance; that is, the values when it is

still mostly carbon and is not largely graphitized. In an arc lamp, the arc of which has a temperature probably even greater than 3,000° C., the ends of the carbon do not seem to graphitize very much; if we may infer from this that they do not graphitize very much in a steel furnace using an arc between carbons, then the constants required for calculating the electrodes for such furnaces should be those of carbon before graphitization. From his curve, Fig. 3, it seems that up to about 1,600° C. there is no very great change in the carbon, hence we may be reasonably safe in assuming that carbon will practically remain carbon within that range.

In one part of his paper (shortly after discussing Fig. 3) he states that the average resistivity of graphite between 3,200° and 200° C. is about 78 per cent. of that when cold, which he had previously stated was 0.00032. This corresponds closely with the figure which I assumed in my paper, after allowing for the lower temperature, namely, 1,700° in my case; I had deduced my value from his own experimental data. Later on, however, he adopts 0.00032 for the mean and uses 0.00035 for the cold resistance. It would be interesting to know why he increased this figure, if the graphite was the same. The difference, however, is not great enough to be very important.

His determination of the mean heat conductivities of graphite and carbon by means of "idlers," is of great interest, as it appears to be the first determination made of mean values for that range of temperature. If correct, the results are of great importance. In his revised paper he has, however, changed them very materially, making them much higher. As first published, his figure for carbon agreed almost exactly with the one which I had assumed; but as I had taken his former results of 1 to 8, while he now gets 1 to 10, his figure for graphite now becomes quite a little higher than mine, which were based on his former ones. It may be of interest in this connection to say that Mr. A. L. Queneau has recently made a determination for carbon and obtained practically the same figure which I had assumed.

Mr. Hansen's figure for graphite seems very high, and I hope it will be redetermined or checked, for if too high it does injustice to graphite. The embedded part of the 4 x 4 electrode in this experiment was only 4¼ inches, which is so short relatively to

the section that the uncertainty in the virtual length becomes quite great; hence I would not like to accept this determination without considerable reserve.

My figure for the electrical resistivity of carbon was deduced from Mr. Hansen's own data for carbon. It differs very greatly from his present one, which, however, is easily explained in that what he here calls carbon is no longer carbon, but an unknown mixture of carbon and graphite. He himself admits that the carbons in his case were partially graphitized; in my opinion he misleads one by still calling the material carbon.

Mr. Hansen, throughout his paper, discourages the use of the so-called "constants" in calculations, on the ground that they are too indefinitely known and too uncertain. It is no wonder that he gets this impression from his figures for what he calls carbon, because it is really an unknown and variable mixture of carbon and graphite. I agree with him that we have not yet any very reliable data for the heat conductivities of either; but now that the need of them and their usefulness in calculations are realized, it is likely that they will be determined. The resistivities are known with reasonable accuracy.

These "constants" which Mr. Hansen has so little confidence in, are merely the "physical properties" of the materials, and to use them for these calculations is only doing the same thing as is done repeatedly in engineering practice, which, in fact, is largely based on such properties, and in many other cases they are not known accurately either, which accounts for the rather large "factors of safety" used in many engineering calculations.

Moreover, as shown in my paper, these two physical properties fortunately enter into electrode calculations in such a form (under the square root sign) that the need of accuracy and constancy in their values is reduced. I also called attention to the fact that the minimum part of the curve is very flat, which also means that great accuracy is not necessary.

Mr. Hansen seems to divide all his C^2R heats by 2. It would be of interest to know why. Why, for instance, should only half the contact heat go into the cooling water?

The loss from contact resistance is, of course, very properly a true loss, but I cannot agree with him that it should be included in such formulas as I gave. That loss is quite as foreign to those

involved in my analysis, as would be the loss in the transformer or the leads, because it is not one of those which determine the electrode proportions. This is shown by the fact that if an electrode is properly proportioned, these proportions would not be altered by the contact resistance, any more than by the loss in the transformer or in the leads, as long as the temperature at the cold end is that used in the electrode calculation. This contact loss should therefore evidently be added to that obtained by the formulas to give cooling water heat, but it should certainly not be included in the terms of the formulas. It is an independent quantity which must be added if one wishes to compare the calculated losses with those found in the heat of the water.

Some of Mr. Hansen's data is unfortunately very crude, hence deductions made therefrom are necessarily also crude. In the table belonging to Fig. 5, for instance, there are very grave inconsistencies in the column of water losses, which he says below "all check to within a few per cent."

The results of his "calculation of losses for titanium carbide furnace electrodes" are unfortunately only very crude, too crude, in my opinion, to disprove the correctness of an analytical method of calculation. Moreover, he has changed the data so radically in the revision of his paper, that one naturally loses confidence in them.

At the meeting I deduced some results from this data based on the length of electrode stated in the paper as it was when it was presented, namely, 27¾ inches, which seems to agree with other descriptions given by him of this furnace. I stated that in my opinion his own results showed that the electrode of this furnace was already too long, although he had concluded from his experimental analysis of electrode proportions (for these same 8 x 8 electrodes), that they were too short. He has now changed this length to 23.6 inches, that is, considerably shorter. This new figure does not seem to agree with dimensions given by him elsewhere of the same furnace (see his Fig. 2.). My deductions, however, still apply, but merely to a less degree. My formulas used with his own constants still show that the high temperature occurred within the walls, for the larger currents, which is what he described elsewhere as having actually occurred. Hence his experiments seem to confirm my formulas, as far as this is con-

cerned. My formulas also indicate that the electrodes for those larger currents were too long, instead of being, as he thought, too short.

In still defending the older "current density" rule for designing electrodes, he states that he knows from experience that when he exceeded 150 amperes per square inch for graphite and 50 for carbon for any considerable period the end wall of his furnace became "badly corroded." By such "corrosion" is apparently meant that the temperature of the electrode within the wall is too high. I maintain that whether this high temperature will exist within the walls or not depends on the relation of the section and length, and not on the current density; and that therefore his deduction concerning current density is not correct. It would probably be very easy to show by experiment that with a *greater* current density this would not occur, and that it would occur with a *less* current density, depending on the relation between length and section; if so, the current density is not the determining factor.

Mr. Hansen finds that the loss for graphite would be about twice that for carbon, based on his figures for those two materials. In this, however, he should have added that his carbon was not the carbon of commerce, but his partly graphitized mixture. If, however, this mixture is so much better for electrodes, which I do not deny may well be the case, then why not make such a mixture for commercial electrodes.

As shown in my paper, however, the minimum loss is not the only criterion. The shape of the electrode is often of more importance. The figures for his partially graphitized carbon show that the electrode would have very awkward proportions, being nearly a cube, according to his own deductions. Hence that alleged gain in economy of power cannot be realized in practice and is only a gain on paper.

His explanation concerning the "check valve" effect, near the end of his paper, is completely anticipated in my paper.

The closing sentence of his paper, before his revision of it, indicates that he did not understand my formulas, as he speaks of H being a minimum and l a maximum, neither of which are ever the case; l can be made anything and is never a maximum, and H is never a minimum unless the length is infinity. Neither

of them are the kind of quantities which have maximum or minimum values in practice. Nor is $H = C^2R$ as he states, but which may be a typographical error.

As he opens his paper with a quotation from a former article of mine, in which I ventured to predict that it would probably be "simpler, quicker, cheaper and more reliable"[1] to determine the laws of the best electrode proportions by calculation than by experiment, I may be permitted to explain here that in saying this I had in mind his own former interesting attempt[2] to determine the best proportions of electrodes by experiments as opposed to calculation, at least as far as length was concerned. In these experiments he described how the wall of his furnace had "repeatedly blown out around the electrode after running a few hours," and that this entailed an expense of approximately $60 per run. This seemed to me to be a rather expensive and slow process of discovering the laws of the best proportions, especially as he concludes from these and other experiments that the electrodes should have been longer, when I feel sure that they were already too long in that furnace, for that section (8 x 8), as is indicated by my formulas. The latter show that the high temperature in that case (with 20,000 amperes) would have occurred within the furnace walls, which was what he seems to have actually found to be the case; hence, if they had been shorter this high temperature point would have been brought to the furnace end. As this would probably have been impracticably short (about 16 inches, using his own constants), it simply means that both length and section should have been increased, but the latter more than the former, so as to get the correct proportions to bring the high temperature point to the hot end where it should be instead of within the wall. His published experiments seem to verify my formulas, as far as it is possible to make calculations with the data he gives, which unfortunately are not always sufficiently complete.

Prof. Jos. W. Richards (*Communicated*): Referring to the data concerning heat conductivity of carbon and graphite in the Appendix to Mr. Hansen's paper, the averaging of the maximum and minimum values of k to get mean values is, in my opinion, unjust-

[1] Electrochem. & Met. Ind., Sept., 1909, p. 402, 2d column last paragraph.
[2] Electrochem. & Met. Ind., August, 1909, p. 359.

tified by the experimental data, considering how they were obtained. The maximum values of k are calculated on the assumption that all the heat generated at the hot end flows through the section under consideration; this may be nearly true for the higher temperatures at the hot end of the bar, but is very far from being a correct assumption for the lower temperatures near the cold end. The minimum values of k are calculated on the assumption that only the heat passing out at the cold end passes through the section under consideration; this may be nearly true for the lower temperatures at the cold end of the bar, but it is very far from being a correct assumption for the higher temperatures near the hot end.

It follows that the values of k most probably correct at the various temperatures are not the arithmetical mean of the maximum and minimum values of k, as calculated by Mr. Hansen, but values of k nearer to the maximum values at the higher temperatures and nearer to the minimum values at the lower temperatures. If we assume that for the highest temperature the maximum value of k is nearly correct and for the lowest temperature the minimum value, and that for intermediate temperatures corresponding intermediate values between the maximum and minimum values, we would have the following probable values of k which I believe more nearly represent the true deductions to be made from the experimental data than the mean values given by Mr. Hansen:

Graphite.

Range of Temperature	k (watt-sec./sec./in./sq. in.)
90-100, av. at 100°	4.76
180-220, " " 200°	3.85
260-340, " " 300°	3.58
350-450, " " 400°	3.38
440-560, " " 500°	3.14
500-700, " " 600°	3.34

Ordinary Carbon.

Range of Temperature	k (watt-sec./sec./in./sq. in.)
37-163, av. at 100°	0.030
105-225, " " 165°	0.113
160-325, " " 242°	0.107
170-330, " " 250°	0.155
200-340, " " 270°	0.183
240-523, " " 382°	0.185
263-543, " " 403°	0.147
283-597, " " 440°	0.115

In calculating the above values of k, I put them forward merely as being more probable deductions from the experimental data than the *mean* values given by Mr. Hansen. My opinion of their reliability is that they are first approximations, and for ordinary carbon are certainly deserving of almost no confidence as reliable data. The range of temperatures is so low as to be almost entirely outside of electric furnace requirements. It would appear to me probable that much more useful and accurate values could be obtained by calculation from actual electric furnace experiments.

MR. HANSEN *(Communicated)*: I do not believe it necessary to reply in detail to Mr. Hering's remarks. A few statements are, however, necessary to make his meaning clear. He probably refers to an unpublished curve shown at the meeting in which an experimentally determined temperature gradient in an electrode was plotted, together with two calculated curves, in one of which latter Mr. Hering's constants were used, in the other those published in this paper. The calculated curves bowed one way, the determined one the other and the difference in the middle of the diagram was some 1400° or 1600° C, the determined temperatures being the low ones. This result I believe also answers other points raised by the same speaker in regard to wall corrosion. As regards the revision spoken of, that was occasioned by a misplaced decimal point in the effective length calculation, which I am very sorry escaped correction in the advance copies.

In reply to Dr. Richards, I wish to state that the heat conductivities are given merely as limits between which the true values lie and may be of some slight interest for that reason. His values are undoubtedly nearer the truth than those he criticises.

A paper read at the Sixteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1909; President L. H. Baekeland in the Chair.

ELECTRODE LOSSES IN ELECTRIC FURNACES.

By E. F. Roeber.

At the Niagara Falls meeting of this Society (May, 1909) Mr. C. A. Hansen presented a paper on some experimental determinations of the energy losses through the electrodes in electric furnaces (*Transactions,* vol. XV, p. 279). Further experimental results on this subject were given by Mr. Hansen in the August and September issues of *Electrochemical and Metallurgical Industry* (vol. VII, pp. 358, 389).

Since that time a method of calculating electrode losses has been given in an article by Mr. Carl Hering in the October issue of *Electrochemical and Metallurgical Industry* (vol. VII, p. 442). The results of Mr. Hering's analysis are also given, rearranged and amplified, in his paper on the "Laws of Electrode Losses in Electric Furnaces," which is published elsewhere in this volume.

A further paper by Mr. Hansen on experiments made by him, with special reference to the application of theoretical calculations, will also be found elsewhere in this volume.

The above named papers of Mr. Hansen and Mr. Hering represent interesting pioneer work and will undoubtedly take an important and permanent place in the literature of electric furnace design.

Since I had made some calculations independently along theoretical lines before I knew Mr. Hering's theory, and since I have come to different results with respect to at least one fundamental point, I will deal in the following paper with this point of difference only. There is no need to state where we agree.

The one fundamentally weak point of Mr. Hering's discussion is, in my opinion, the uncertainty in which we are left with respect to the conditions under which his formulas hold true. This refers to his assumptions concerning the variation of thermal and electric conductivities of the electrode material with temperature.

It is easy to prove that the theory *as developed* is correct only if both conductivities are assumed to be constant. First, with respect to *electric* conductivity, W is defined as the "total heat flow in watts generated in the rod by the electric current; that is, the total C^2R heat." This is then divided by the length of the electrode, giving "w = this C^2R heat per inch of length." This value w is inserted in the differential equation and the C^2R heat per inch of length is treated as though it had the same value at all points of the length direction of the electrode, along which the temperature changes from point to point. Obviously, this whole procedure has meaning only if the electric resistivity is assumed to be constant and independent of the temperature.

With respect to *heat* conductivity, it is to be said that in Mr. Hering's present paper, the differential equation (11) is correct in general. But in differentiating it and thus deriving the fundamental equation $d^2t/dl^2 = w/jkS$, k is treated as constant; and the same is done in the two integrations which follow and which lead to equations (12) and (13).

We are thus forced to conclude that the theory, being developed as a consequence of equations (12) and (13), is based on the assumption that both the electric and heat conductivities of the electrode material are *constant* and independent of the temperature.

It is, of course, generally recognized that the two conductivities vary greatly with the temperature. Under the conditions of operation of Mr. Hansen's titanium carbide furnace, the heat conductivity of graphite at the cold end may be something like 270 times the heat conductivity at the hot end, and the electric conductivity at the cold end something like 60 per cent. of that at the hot end.[1] Confronted with such a condition, we are forced to ask: How much faith can we have in a theory that is based on the assumption of constant conductivities?

Is the theory as developed, wholly of academic interest only, or to what extent can we use it safely in practice? This is a pertinent question and one which requires an answer. The only way of giving a satisfactory answer is to extend the theory by "rigid analysis" to the general case in which the heat and electric

[1] These figures were kindly given to me by Mr. Hansen and are stated to be only approximate. I wish to use this opportunity to thank Mr. Hansen for his courtesy in giving me much interesting information in the course of this investigation.

conductivities are assumed to vary with the temperature. This is the object of the following paper.

The problem can be attacked in two different ways, both of which we will follow.

The first way starts, like the classical mathematical theory of heat, from the investigation of an infinitely small particle. We investigate what happens there and establish the fundamental differential equation. This is then integrated and the results relating to the whole electrode are thus found. This is the way in which Mr. Hering proceeds, treating the conductivities as constants. We will treat them as variables; therefore not only our fundamental differential equation, but the equations resulting from integrations differ from Mr. Hering's equations. We will restrict our theory essentially to the case in which no heat flows out of furnace into the electrode, the electrode end being at exactly the same temperature as the furnace charge next to it, so that no heat flows through the hot electrode end in either direction. In our treatment of this problem, we will first assume that the conductivities are linear functions of the temperature (§ 1), and we will then extend the theory to the case in which the conductivities are any functions whatsoever of the temperature (§ 2). Mr. Hering's equation (5) in his present paper then appears as the most special case of our general formulas.

The second method does not make any use of calculus. We consider the electrode at once as a whole and can without much difficulty derive some general conclusions (§ 3).

§ 1: *Conductivities assumed as linear functions of temperature.*

The following theory is first based on the assumption that the heat conductivity c_h (in watt seconds passing through 1 sq. cm. cross section, for a temperature drop of 1° C. for 1 cm. distance, in 1 second) and the electric conductivity c_e (the reciprocal of specific electric resistance in ohm-centimeter units) are linear functions of the temperature T:

$$(1) \qquad \begin{cases} c_h = k\ (1 - \alpha\ T) \\ c_e = c\ (1 + \beta\ T) \end{cases}$$

We use the minus and plus sign before α and β respectively, because according to Mr. Hansen, the thermal conductivity

decreases with the temperature, while the electric conductivity increases, both for carbon and graphite, which is quite unusual and interesting.

We will not attempt to give the general solution of the problem of heat flow of a current-carrying electrode under this assumption, but will simply investigate the question: What condition must be fulfilled so that no heat passes in either direction through the hot end of the electrode? For this is, after all, the condition under which electrodes are preferably operated and which should form the basis of electrode design.

The mathematical formulation of this condition is obviously that for $x = 0$ (the hot end of the electrode) the differential quotient dT/dx of the temperature must be zero. By inserting $dT/dx = 0$ for $x = 0$ into the general formulas, we get the particular solution for the case in which no heat flows in either direction through the hot electrode end.

We assume an electrode of uniform cross-section and assume that no heat is given off laterally to the refractory walls. We further assume that the temperature at any point does not change with the time (stationary condition) and that at any moment it is the same in all points of a cross-section perpendicular to the length direction. We make this length direction the X-axis of ordinates, the electrode extending from $x = 0$ to $x = l$ (measured in cm.) then the temperature T at any point x of the electrode depends only on x.

If s is the cross-section of the electrode (in sq. cm.) we will investigate what happens within the infinitely small space between the cross-sections at the distances x and $x + dx$ from $x = 0$.

As c_h is the heat conductivity, the heat H_1 passing per second through the first surface into our assumed space is $- s\ c\ dT/dx$. The heat H_2 leaving our assumed space through the second surface is $- s\ c_h\ dT/dx - s\ d/dx\ (c_h\ dT/dx)\ dx$. The Joulean heat H_2 produced within our assumed space from electrical energy per second is $i^2 dx/c_e s$ where i is the current in amperes and c_e the electric conductivity. (All three heat quantities are given in watt seconds). Hence the heat $H_1 + H_3$ enters our space, H_2 leaves it, so that as the temperature does not change in time, $H_1 + H_3 = H_2$ or

$$(2) \qquad \mathrm{d}\left(c_h \frac{\mathrm{d}T}{\mathrm{d}x}\right) = -\frac{i^2}{s^2\, c_e}\,\mathrm{d}x$$

By means of the substitution $c_h\, \mathrm{d}T/\mathrm{d}x = y$, we get

$$(3) \qquad y\,\mathrm{d}y = -\frac{i^2}{s^2}\frac{c_h}{c_e}\,\mathrm{d}T = -\frac{i^2}{s^2}\frac{k}{c}\frac{1-\alpha T}{1+\beta T}\,\mathrm{d}T$$

By integrating and determining the constant of integration in such a way that $\mathrm{d}T/\mathrm{d}x = 0$ for $T = T_0$ $(x = 0)$ we get

$$(4) \qquad \frac{\mathrm{d}x}{s} = -\frac{1}{i}\frac{(1-\alpha T)\,\mathrm{d}T}{\sqrt{\frac{2}{kc}\frac{\alpha}{\beta}\left\{T - T_0 + \left(\frac{1}{\alpha}+\frac{1}{\beta}\right)\lg\frac{\beta T_0 + 1}{\beta T + 1}\right.}}$$

By integrating between the limits $x = 0$ and $x = l$ ($T = T_0$ and $T = T$ respectively), we get

$$(5) \qquad \frac{l}{s} = \frac{1}{i}\sqrt{\frac{kc}{2}}\int_{T_1}^{T_0}\frac{(1-\alpha T)\,\mathrm{d}T}{\sqrt{\left(\frac{1}{\beta}+\frac{\alpha}{\beta^2}\right)\lg\frac{\beta T_0 + 1}{\beta T + 1} - \frac{\alpha}{\beta}(T_0 - T)}}$$

This is the general solution of our problem for the case that both conductivities are linear functions of the temperature. It gives that ratio of length to cross-section, for which, with a current i and the temperatures T_0 and T_1 at the two ends, no heat will flow in either direction through the hot end of the electrode. The integral on the right hand can be evaluated by graphical means; it depends only on α, β, T_0 and T_1 and nothing else.

If in equation (5) we make $\alpha = 0$, we get the solution for the case in which the heat conductivity is a constant, independent of temperature, while the electric conductivity is a linear function of temperature (temperature coefficient β). It is

$$(6) \qquad \frac{l}{s} = \frac{1}{i}\sqrt{\frac{kc}{2}}\int_{T_1}^{T_0}\frac{\mathrm{d}T}{\sqrt{\frac{1}{\beta}\lg\frac{\beta T_0 + 1}{\beta T + 1}}}$$

The integral on the right hand can be evaluated graphically; it depends only on β, T_0 and T and nothing else.

If in equation (5) we make $\beta = 0$, we get the solution for the case in which the electric conductivity is a constant, independent of temperature, while the heat conductivity in a linear function of the temperature:[2]

$$(7) \quad \frac{l}{s} = \frac{1}{i}\sqrt{\frac{kc}{2}} \int_{T_1}^{T_0} \frac{(1 - \alpha T)\, d\, T}{\sqrt{(T_0 - T)\left(1 - \frac{\alpha}{2} T_0 - \frac{\alpha}{2} T\right.}}$$

$$= \frac{1}{i}\sqrt{2\, kc\, (T_0 - T_1)\left(1 - \frac{\alpha}{2} T_0 - \frac{\alpha}{2} T_1\right)}$$

Since $k\,(1 - \alpha/2\,(T_0 + T_1))$ is the arithmetic mean value of the thermal conductivity between T_0 and T_1, we find by comparison with the following equation (8) that for the determination of the best ratio of l to s it is correct to use the formula (8) which holds good for constant conductivities independent of the temperature, if we insert in it the arithmetic mean value of the thermal conductivity. But this is true only if the thermal conductivity is a linear function of the temperature and *if the electric conductivity is independent of the temperature.*

Finally, if we make $\beta = 0$ in equation (6) (by employing again the same series for lg), or if we make $\alpha = 0$ in equation (7) we get the solution for the case in which both the electric and heat conductivities are constant. It is

$$(8) \quad \frac{l}{s} = \frac{1}{i}\sqrt{\frac{kc}{2}} \int_{T_1}^{T_0} \frac{d\, T}{\sqrt{T_0 - T}} = \frac{1}{i}\sqrt{2\, kc\, (T_0 - T_1)}$$

This is identical with Mr. Hering's equation (5) in his present paper or his equation (22) in his article in the October issue of *Electrochemical and Metallurgical Industry:*

[2] The calculation is best carried out by making use of the series

$$\lg \frac{\beta T_0 + 1}{\beta T + 1} = \frac{\beta (T_0 - T)}{\beta T + 1} - \frac{1}{2} \frac{\beta^2 (T_0 - T)^2}{(\beta T + 1)^2} + \frac{1}{3} \frac{\beta^3 (T_0 - T)^3}{(\beta T + 1)^3} - * * * * *$$

This is correct if $\beta^2 (T_0 - T)^2 / (\beta T + 1)^2$ is smaller than unity, so that this series is suitable for finding the limit of the various terms in (5) and (6) for $\beta = 0$ This same series may be used to find approximate formulas for the integrals in (5) and (6), if such approximate formulas should be preferred to the graphical evaluation of the integrals suggested above

$$\frac{S}{L} = 0.3456\ C\sqrt{\frac{r}{k\,T}}$$

The only difference is in notation and units employed. Mr. Hering writes S for our s, L for our l, C for i, r for $1/c$, T for $T_0 - T_1$. He measures the lengths in inches and areas in square inches; we employ centimeters and square centimeters respectively. This difference would make no change whatever in the formulas.

Further, Mr. Hering measures the heat conductivity in calories, etc., while we measure it in watt seconds. This accounts for the difference in the numerical constants in the two formulas.

§2. *Conductivities assumed as any functions of temperature.*

The above theory shows clearly that Mr. Hering's present formula (5)—our equation (8)—is valid only for the ideal case of *constant* heat and electric conductivities independent of the temperature. For the more general case of both conductivities being linear functions of the temperature, "rigid analysis" does not lead to Mr. Hering's simple formulas, but to the more complicated formula (5).

It would be easy enough to make a numerical calculation on the basis of formula (5), if it were really true that both conductivities are linear functions of the temperature; but from Mr. Hansen's figures we are hardly justified in drawing that conclusion.

My chief reason for publishing the above mathematical theory is the ease with which it can be generalized so as to apply to the most general case that the heat and electric conductivities are any functions (more or less complicated) of the temperature. Instead of (1) we have now

$$(1^a) \qquad \begin{cases} c_h = f^1\,(T) \\ c_e = f^2\,(T) \end{cases}$$

where f^1 and f^2 represent any functions whatsoever. We assume nothing regarding the nature of these functions. We only assume that c_h and c_e depend in a distinct way on the temperature T.

In our fundamental differential equation (2) nothing is changed. Instead of (3) we get now

$$(3^a) \qquad y\,dy = -\frac{i^2}{s^2}\,\frac{f^1(T)}{f^2(T)}\,dT.$$

By integrating and writing for the sake of brevity $g(T) = \int \frac{f^1(T)}{f^2(T)}\,dT$ and by determining the constant of integration in such a way that $dT/dx = 0$ for $T = T_0$, we get instead of 4:

$$(4^a) \qquad \frac{dx}{s} = -\frac{dT}{i\sqrt{\frac{2}{c_h^2}\{g(T_0) - g(T)\}}}$$

By integrating between the limits $x = 0$ and $x = l$, ($T = T_0$ and T_1 respectively), we see that the integral on the right hand depends on nothing but T_0, T_1 and on the constants which determine c_h and c_e as functions of T according to (1^a). We will express this by writing $f(T_0, T_1, c_h, c_e)$ for the evaluated integral at the right hand. Our final result is

$$(5^a) \qquad \frac{l}{s} = \frac{1}{i}\,f(T_0, T_1, c_h, c_e)$$

For the most general case, in which the thermal and electric conductivities are any functions whatsoever of the temperature, we have thus proven the following fundamental principle:

If no heat passes in either direction through the hot electrode end within the furnace, while the current i passes through the electrode, the ratio of length to cross-section equals the reciprocal value of the current i, multiplied by a value which depends only on the electrode material and the two temperatures T_0 **and** T_1 ***at*** *the two ends of the electrode.* The only two assumptions made are the condition of stationary operation (the temperature being independent of the time) and the condition that no heat is given off by the electrode laterally to the refractory walls.

As we have seen, the calculation of the value of f in (5^a) leads to mathematical complications even in the simple case that the conductivities are linear functions of the temperature. Further, if we had the exact formula for $f(T_0, T_1, c_h, c_e)$, in order to calculate it, we would first have to determine experimentally how the thermal and electric conductivities change with the temperature within the whole temperature range from T_0 to T_1.

It seems to me very much simpler to determine instead directly by experiment the value of $f(T_0, T_1, c_h, c_e)$ itself by making use of the formula (5^a) as will be shown later on.

§3. *Discussion without use of the calculus.*

We will now investigate what general conclusions can be drawn, without making any use of differential calculus, by considering at once the electrode as a whole.

We assume nothing concerning the variation of the heat and electric conductivities of the electrode material with the temperature. We only assume that the condition of operation is stationary (the temperature at any point remaining constant in time) and that no heat is given off from the electrode to the surrounding walls. Compared with the preceding part of this paper, the following investigation is more general in so far as it covers any stationary operation of the furnace, and not only the special case in which no heat is flowing in either direction through the hot electrode end within the furnace.

Under our assumed conditions, the heat A (positive or negative) flowing from the furnace inside through the hot electrode end into the electrode, and the heat B (always positive) flowing from the electrode through the cold end to the outside (for instance, to the waterjacket) have distinct values, which depend on the special conditions under which the stationary operation of the furnace takes place.

It is then always possible to write A and B as the difference and sum respectively of two other values x and y.

$$A = x - y$$
$$B = x + y$$

This involves, of course, no assumption at all. It is so far simply a play with mathematical symbols. But it is easy to see that x and y have a distinct physical meaning.

By subtracting A from B we get,

$$y = \frac{B - A}{2}$$

B — A is the difference of the heat which flows out of the electrode at the cold end and the heat which flows into the

electrode at the hot end. Since according to our assumption no heat flows through the side walls of the electrode, B — A must be the total Joulean heat generated within the electrode. Hence y = one-half the total Joulean heat.

Further, it is evident that $x = (A + B)/2$ = half the sum of the two heats flowing into the electrode at the hot end and out of the electrode at the cold end (both measured in the same direction). Hence we see that we have a superposition of the two phenomena: First, a flow of the same heat quantity $x = (A+B)/2$ through the two ends in the same direction; and, second, a generation of Joulean heat $2y$ within the electrode from electrical energy, this heat flowing off impartially one-half $(+y)$ to one side and one-half $(-y)$ to the other side.

So far everything is very simple. But the trouble is that in the general case we cannot consider the two phenomena independently of each other. The heat x is not "the heat which would flow if there were no current." Both the temperature drop from the hot inside to the cold outside end and the generation of Joulean heat are responsible for the temperature gradient within the electrode. Hence the Joulean heat affects the temperature and therefore the heat conductivity at any point so that the heat flow x is not the same as though no current were flowing, because in the latter case the temperature and the heat conductivity at any point would have different values from our case.

In quite the same way the Joulean heat $2y$ is not the same as though there was no temperature drop from the hot to the cold end, because the latter affects the temperature gradient and therefore the electric conductivity at any point, and therefore the Joulean heat.

Hence the two phenomena of simple heat conduction x and Joulean heat production $2y$ are not independent of each other. We see, however, that whatever may be the function which determines the dependence of the two conductivities on the temperature, the condition under which no heat passes through the hot electrode end in either direction is given by the equation $x = y$. Or, in other words, the heat flow x must equal half the Joulean heat.

In view of the mathematical difficulties which arise if we try

to find the exact formulas for x and y, it seems best to stop here with the analytical discussion of the problem and to rely upon experiment for further developments. This course has been followed already both by Mr. Hansen and Mr. Hering.

Mr. Hansen in his paper, published elsewhere in this volume, analyzes with great skill the electrode losses in one of his furnaces on the basis of the equation $B = x + y$, or electrode loss = conducted heat, due to temperature difference, + one-half Joulean heat.

He calculates the two losses on the basis of mean values of electric resistivity and heat conductivity under the conditions of his furnace operation. His definition of "effective electrode length" and the way in which he handles contact resistance in his analysis are valuable contributions to the solution of our problem.

Essentially the same idea of supplementing the theory by experiment is the basis of Mr. Hering's paper on "A New Method of Measuring Mean Thermal and Electrical Conductivities of Furnace Electrodes," also published in this volume. The experimental arrangement of his method of measurement, as illustrated in Figs. 1, 2, 3 in his paper, is as ingenious as simple.[3]

His formula (3) for measuring the mean electric resistivity under the conditions of the experiment is free from any objection. As to the general applicability of his equation (1) for determining the mean heat conductivity some doubt may be raised, because this equation is based on the assumption of constant thermal and electric conductivities. It is, however, easy to make Mr. Hering's really pretty method free from any objection, by basing the measurement on our equation (5^a) which was proved above to be true whatever may be the nature of the relation which gives the conductivities as functions of the temperature.

In this way we get directly the best ratio of cross-section to length of an electrode, while Mr. Hering determines the two

[3] The heat insulation is certainly as perfect as can be imagined. The electric insulation of the test rod in the centre from the surrounding heat insulating rods is less perfect if all the rods are connected in series as suggested. In this case the potential difference between the test rod in the centre and one of the surrounding rods will be six or eight times the potential difference between the ends of the test rod. At the existing high temperature of the experiment, this potential difference might cause a leakage current from the centre rod to the outside rod. This would, of course, spoil the results. The remedy would be simply to connect all rods in parallel. Then the electric insulation is as perfect as the heat insulation. But special precautions would be necessary to have the current exactly the same in all rods, and this might cause complications. Only experiment can show whether this criticism is well founded.

mean conductivities and calculates the above ratio from them. That our method does not give the mean conductivities is no disadvantage, since the mean conductivities as determined by this method are mean values under the condition of electrode operation (so that, for instance, the mean heat conductivity thus found is different from the mean heat conductivity in simple heat conduction). Practically the only application of the mean conductivities as found by Mr. Hering's method, would, therefore, be to determine the best ratio of length to cross-section.

It seems, therefore, just as good or even simpler to determine the latter ratio directly by Mr. Hering's method on the basis of our equation (5^a) as follows:

Our equation (5^a) is based on the assumption that $dT/dx = 0$ at the hot end of the electrode. As explained in Mr. Hering's paper, if an electric current of i amperes is passed through the rod (length $2l$ centimeters, cross-section s sq. centimeters), in Fig. 1 of his paper, while the ends a and b are kept at a constant temperature T_1, the temperature of the rod will be raised and will be a maximumm T_0 at the centre m. Hence $dT/dx = 0$ at the centre, and we can apply our equation (5^a) directly to either half of the electrode from the centre to one of the ends.

Our equation (5^a) states that li/s is a value which depends only on T_0, T_1 and the electrode material.

Assume that we make a test with graphite. We will use a rod ab of length $2l$ and cross-section s. We keep the temperature T_1 at the two ends a and b constant, for instance, by water-cooling, and we measure the temperature T at the centre and the current i. Then according to our theory li/s is a function of T_0 only.

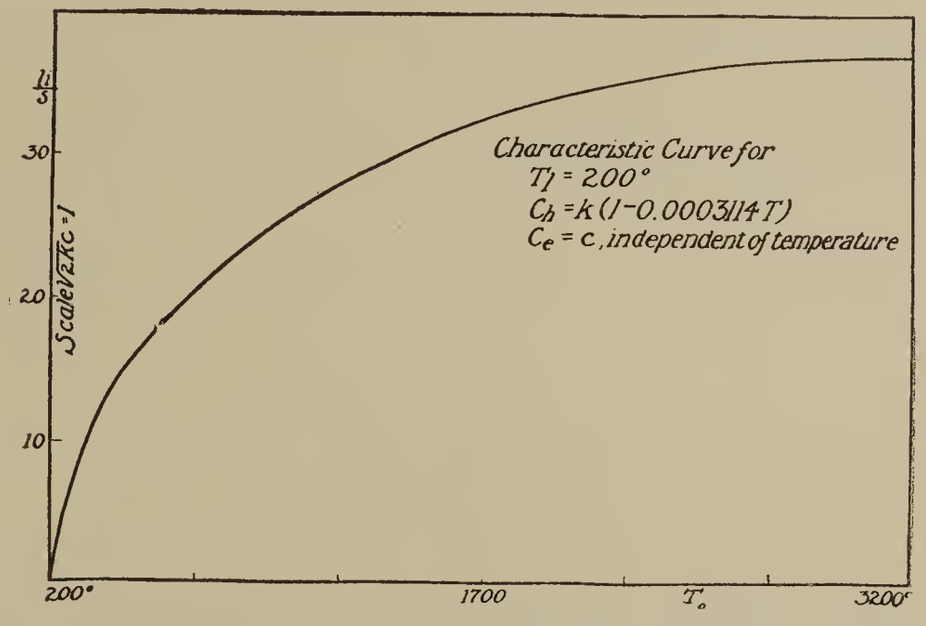
By raising the current i from zero upwards, and waiting each time until equilibrium is established, every increase of the current i will produce a rise of temperature T_0. We can then prepare a table or plot a diagram with the temperatures T_0 as abscissas (T_0 always greater than T_1) and the value li/s as ordinates.

Our equation (5^a) states that if we repeat the same experiment with another rod of length $2l'$ and another cross-section s' and if the current which produces the maximum temperature T_0 at the centre is now i', the value $l'i'/s'$ for any temperature T_0 must be the same as in the preceding set of experiments. This would

be the most direct and crucial test of the correctness of the theory and the proper execution of the measurement.

When this is done, the diagram we have plotted will directly answer the question of the best ratio of length to cross-section to be selected for any graphite electrode with water-cooled end (temperature T_1 fixed), if the temperature T_0 of the furnace and the current i are specified. We simply find from the curve in the diagram the value li/s which corresponds to the given temperature T_0, and by dividing the value li/s by the given current i we find the best ratio of l to s.

Of course, the diagram will answer likewise the question what



Curve giving relation between $\frac{li}{s}$ and T_0

current i would have to be employed with an electrode of given length l and cross-section s in order to use it under best conditions (so that no heat flows in either direction through the hot end) for a given temperature T_0 at the hot end.

Any characteristic curve of this kind holds good only for the same material and for the same temperature T_1 of the cold end. For any other material and for any other temperature T_1 of the cold end other characteristic curves must be plotted.

To give a rough idea of the general character of such a curve, we give in the adjoining diagram the theoretical curve, based on our equation (7), for an assumed material which (like graphite, according to Mr. Hansen) has at 200° a thermal con-

ductivity 270 times that at 3200°; we assume for our material that the thermal conductivity is a linear function of temperature. This gives

$$c_h = k\ (1 - 0.0003114\ T)$$

We further assume that the electric conductivity $c_e = c$ constant and independent of temperature. The cold end of the electrode is maintained at a constant temperature of 200°. The abscissas give the temperature T_0 at the hot end, while the ordinates give the value li/s. Our scale of ordinates is fixed by the assumption $\sqrt{2kc} =$ unity. In practice l would be given in inches (or centimeters), s in square inches (or square centimeters), and i in amperes. This would change nothing but the scale of ordinates.

It seems decidedly worth while to determine experimentally such characteristic curves for different cold-end temperatures T_1 and for different materials, especially graphite and carbon. When these curves have once been determined, they will give immediately all information necessary for practical purposes, and in the simplest form.

DISCUSSION.

MR. CARL HERING: I agree with Dr. Roeber, as will be seen in my own paper, that the premises made in my paper were not all strictly correct, but were only approximations, hence any criticism that I did not know this is hardly fair. The real question is, how great is the error. No one has yet determined that.

As to the other matter to which he refers, I am about to read a paper, the next one on the program, which shows that a more correct way is to determine the constants under the same conditions of temperature under which they will be used in the formulas; they will then be right, as the error referred to is then balanced by an equal error in the opposite direction.

CARL HERING *(Communicated)*: If the modest attempt made in my paper to obtain a first approximation to more rational methods of proportioning furnace electrodes, than those heretofore in use, brings out such interesting and valuable papers as

this one by Dr. Roeber, and more accurate determinations of those important physical properties of materials, I shall feel well satisfied that my efforts have not been wasted, even if my first approximations to simple formulas, should turn out to be so very wide of the mark as some of my critics try to make it appear, but which I feel sure will not be the case.

Dr. Roeber's analysis is very interesting and, in some respects, of considerable value. I was disappointed only in that the results do not appear to be of such direct practical application (as I had hoped they would be) so as to answer the real issue raised in the discussion of my paper, namely, how much in *actual figures*, the proportions obtained by my approximate formulas differ from those calculated by a more exact but far more intricate method. That is, whether the point raised by my critics was a mountain or a mere mole hill. Nor is his chief formula even in such a form that values can readily be substituted in it to calculate the more accurate results.

Notwithstanding its failure to help us to get better practical results than by my approximate formulas, his analysis is of great interest from the academic standpoint. I had tried the integration on similar lines many months ago, under the assumption of rectilinear functions, but abandoned it as it seemed (what now appears evident from Dr. Roeber's formulas) that the results, even if the integration were possible, would probably be too cumbersome for practical use, especially for furnaces, which unlike dynamos and other exact machinery, cannot be constructed and operated under exact conditions; hence it is of no real importance to be able to design them with great accuracy and precision.

In making my analysis I was striving for practical results; the analysis was only incidental. I was not satisfied with the former rules based, as they were, on current densities, lowest possible resistances and the summation of the two losses. Dr. Roeber's analysis certainly shows that my approximate results are at least much nearer to the exact ones than those based on the older rules would be.

Aside from the fact that Dr. Roeber extends and confirms the more important of my results in a number of respects, the substance of his adverse criticism of my paper is briefly as follows. That my proof is limited to constant conductivities while in the

premises it was assumed that the heat conductivity may vary by a straight line law; hence the proof did not cover the complete premise. In this he is of course correct and with the permission of those who pointed out this inconsistency, I have in the revised paper reduced the scope of the premises to fit the proof. The proof is now rigid and the laws given are absolute under the premises assumed. The formulas and laws are now approximate only in so far as the premises differ from the conditions in practice. I will explain in my reply to the discussion of my paper how this inconsistency occurred. Briefly, it was that other calculations made many months ago, showed that my formulas were quite correct when the heat conductivity varied in this way; I therefore added this to the premises after the paper was written, last June, but forgot to add my reasons for so doing.

But now Dr. Roeber has himself furnished the rigid proof of that case, thus showing that what was said in my paper before revision was really correct but was simply not proved as to this specific case. I am glad he supplied this rigid proof that my original formulas applied also to such a variation of the thermal conductivity, because the calculations which had convinced me were only an indirect proof and not a rigid mathematical one. I did not think then that the possible error would be important enough in practice to warrant the intricate calculating necessary to determine it quantitatively. He has now shown that this error is zero. His proof of this point is the solution of his equation (7) in which he finds that the correct value for the heat conductivity is the mean or average for that range of temperature, which is the value assumed in my formulas.

But in his oft repeated statement that the conductivities in my formulas are "independent of the temperature" he is quite wrong, as I stated clearly and distinctly in the paper that the values used are the *means for the given ranges of temperature.* Hence they are decidedly not "independent of the temperatures." The values which were specified would clearly be different for different ranges of temperature, hence they certainly are not independent of the temperature. It is true that the values are assumed (in my proof) to be the same from one end of the electrode to the other, and in that limited sense they are not supposed to change in different parts of that electrode. But by taking for

this constant value, the *average* for the specific range of temperature of any particular case, as specified in the paper and as is so often done in similar problems in practice (and even in academic calculations, when approximate results are sufficient), I did not believe and do not now believe the error (which was admitted in assuming those premises) would be important when both conductivities varied. For a straight line variation of the heat conductivity at least, he himself has shown the error to be absolutely zero.

In the discussion at the meeting, my critics gave the impression that my formulas were of little or no value, because I had used *mean* or *average* values for the varying ones, and Dr. Roeber in this paper throws grave doubts on the results by questioning whether we could have any "faith" in a "theory which is based on the assumption of constant conductivities," a question which, by-the-way, he still leaves unanswered, as he gives no quantitative results. Nobody at the meeting could show at the time, nor have they shown since, that my formulas were thereby vitiated; nor even that the error in this approximation was great. The latter is not even shown in Dr. Roeber's interesting paper, although it might, perhaps, be determined from it by someone who had the patience to work out the final quantitative results from his formidable and only partially solved (unintegrated) formula (5).*

In view of that discussion, it is somewhat amusing that it now turns out from the present paper that at least as far as the heat conductivity is concerned (the only one referred to in the premises and apparently the more important one), there is no error at all, for a straight line variation. Whether or not there is any appreciable error in also assuming the electrical resistivity to vary according to a straight line law, remains to be shown by actual figures. The present indications are that the error is not great either. If, as Dr. Roeber says, the heat conductivity variation is according to Mr. Hansen as great as 270 fold, while the electrical conductivity varies only about 50 per cent., it would seem that by far the greater of the two assumed errors had been disposed of by Dr. Roeber's proof that it was zero.

Dr. Roeber starts out with an assumption which I had to prove

* See discussion by Dr. H. C. Richards, page 304, received since this was written.

first, as it was certainly not general knowledge when I first stated it at our May meeting, at which it was met with some ridicule. This assumption is that an electrode should be so proportioned that there is little or no flow of heat through the hot end. It was apparently considered to be little short of heresy in May of this year, to suggest that the resistance loss be actually increased intentionally, in order to reduce the total loss. Prior to this the general rule for designing electrodes was to make the resistance as low as practicable and to use current densities as the determining factor. What was heresy six months ago, is now assumed as "obvious;" such is the progress of our time. I shudder to think of the mathematics required to prove the general case in the thorough way Dr. Roeber has done, by starting with nothing more than what I had to start with originally, not to mention the prejudice I had overcome in condemning the older methods.

Dr. Roeber has not proved that his assumption corresponds to the case of minimum total loss, when the variations of the conductivities are considered. Probably it is so, or at least nearly so, but as he has not proved it, his proof is not complete either. My starting point was to find out under what conditions the loss was a minimum; he assumes that condition and proceeds from there on.

Dr. Roeber also had the advantage of my statement last May that the proportions are independent of the actual size, being governed only by the ratio of the length and section. Hence he could at once start to determine his ratio. I had to prove this first, as it was not general knowledge then.

I mention these points because in his bibliography of the literature on this subject at the beginning of his paper, he omitted all reference to my remarks at our May meeting† in which the general results of my analysis were announced, although briefly, including the figures 1, 3, 4 and 5 of my present paper, almost exactly as reproduced now.

Dr. Roeber's method, moreover, does not show as clearly and directly some of the other consequences of these relations, which even though they may be only approximate, are nevertheless of interest and value as approximations. One of these is the so-

† Vol. XV, p 300-2.

called "electrode voltage," a new and presumably valuable characteristic property of electrode materials.

Dr. Roeber lays considerable stress on a supposed "uncertainty" in the assumptions in my paper. I do not see that there is any uncertainty whatsoever. It was distinctly stated several times that the conductivity values were the *means* of all those for the given temperature ranges. Whenever a mean or average value of a large number of different values is used in place of the latter, as is so often done in engineering practice, it means that this value is assumed to be a constant in the discussion, and it was therefore so assumed in my algebraic proof. This left no uncertainty. I admit only that the reasons for having extended the premises to include variable heat conductivity, had been omitted by oversight, causing an inconsistency which has now been corrected; it was not an uncertainty. Concering the electrical resistivity, no uncertainty could have arisen, as it was clearly stated that the assumed value was an average or mean for the particular range. In the earlier publication of a large part of my paper, referred to by Dr. Roeber, the straight line law for the electrical resistivity was by error included in the premises, but in the present paper now under discussion this was not the case. It is not admitted thereby that the error is serious; because if it exists at all, it is probably quite negligible in practice; but it was not consistent with the term "absolute" used with respect to the laws deduced. They may be only approximate instead of absolute, for a varying electrical conductivity. Nobody has yet proved it one way or the other.

Dr. Roeber is also guilty of a slight and unimportant inconsistency when he states that my resuits are strictly correct only for constant conductivities and then proves them to be correct also for a thermal conductivity varying according to a straight line law.

I cannot agree with him that the "only" way to solve this problem of varying conductivities is by a "rigid" analysis. I believe the way I recommeded in the paper is even better than analysis, namely determining the correct "mean" values directly by experiment by means of the same formulas in which they are afterwards used. He himself recommends this very method at

the end of his paper, only that he uses a slightly different scale for the ordinates, which does not change the curve.

That the electrical conductivity of carbon and graphite increases with temperature, was known many years ago, notably in connection with incandescent lamps, and was therefore not discovered by Mr. Hansen as Dr. Roeber seems to believe.

Although Dr. Roeber lays great stress on the error due to assuming average values for the conductivities, he includes in his proof the assumption of perfect heat insulation. It is very probable that the inaccuracy involved in the latter assumption is by far the greater of the two; if so, his interesting formula is still only an approximate solution of the complete problem and is only a step closer than mine.

When Dr. Roeber says that it is "easy enough to make a numerical calculation on the basis of formula (5)," which is the unintegrated one, I do not believe many engineers engaged in practical work will agree with him.

The short and simple proof which he gives in the latter part of the paper is very neat and interesting. It is especially so, if it should be true that the heat x is that which would flow when there is no current, but when the temperatures along the electrode are those which existed under the normal conditions while current was flowing. That is, if x represents the flow of furnace heat during the first infinitesimal moment after the current is stopped. If this is true, and if it is also true in general that for no flow through the hot end, the C^2R heat is always equal to twice the flow of furnace heat, then it seems possible to prove by a simple graphical method, that variable conductivities in place of constant ones, would change only the heat gradient between the ends but would not alter the end results; that is, the simple formulas would apply without any error, to the electrode as a whole, and would be approximate only when applied to the interior parts of the electrode between the ends. As it is almost always the electrode as a whole which is of importance in practice, it would mean that the formulas are correct for almost all cases occurring in practice, under the assumed premise of perfect heat insulation.

The experimental determination of that curve at the end of his paper, is exactly what was proposed in my second paper, only that he uses a different scale of ordinates. This would not

change the curve. Generally all that is wanted is a single value for the particular range of the furnace which one is designing; hence it is then unnecessary to construct the curve. If, however, it is desired to give all the values for all possible ranges, then the curve becomes necessary, as it takes the place of a table of different values. I still think, however, that the ordinates had better be made the mean conductivities and not the relation of three quantities as he suggests.

Concerning his most general formula in which the equations for the varying values of the conductivities are to be introduced, I desire to call attention to far more serious results the academician may thereby lead us to, than neglecting small errors in formulas which at best need be only approximately correct. The conductivities are not true variables in the broad sense that they may have any values in a given problem, theoretically from zero to infinity, like the length, cross section, loss, etc. The conductivity values have limits; but an algebraic equation of their variation with the temperature, does not take notice of such limits, and purely algebraic deductions from such expressions may therefore give results which are outside of those limits and hence are absurdities, even though they are mathematically rational.

His very general formula (5a) is exactly like my (5) except that in the latter the function is a specific one, instead of a general one. Hence the latter is rigidly correct in its construction and in the quantities involved, the only question being whether the particular function is exactly the square root, and whether r, k and T have those relative relations to each other.

Concerning the point which is of most importance to the practical engineer, namely the magnitude of the errors introduced by using average or approximate, instead of very accurate values for the conductivities, I here call attention to two facts shown by my analysis, namely that in the two important formulas for calculating electrodes, the conductivities both occur under the square root sign, which means that any inaccuracy in their values affect the final result less. Also that the curve of losses is quite flat at the minimum point, which means that inaccuracies in the final proportions affect the loss very little, provided one is somewhere near the desired minimum point.

His suggested experimental test of his theoretical results, by

means of the method described in my other paper, might give very favorable results and yet the theory may be only approximate when applied to actual cases, because in that method, what seems to me to be the largest error in his premises, is not eliminated, namely that due to the imperfect heat insulation. In that method the heat insulation would probably be nearly perfect, much more so than in practice. Hence this way of checking might confirm his algebraic deductions but would not necessarily mean that his more complicated formulas are much nearer to the actual results than my simpler ones.

Dr. E. F. Roeber (*Communicated*): While I am certainly obliged to Mr. Hering for his detailed criticism, I will restrict my reply to a discussion of two points of general interest—the most suitable starting point of the analysis of electrode losses, and the way in which the results are changed by taking heat leakage from the electrode into the walls into account. First, I must answer, however, a personal remark of Mr. Hering.

Mr. Hering complains that in my introductory references to former literature I omitted all reference to his remarks at the May meeting (Vol. XV, pp. 300-2), in which, as he now says, the "general results of his analysis" were announced. Reference to these remarks will show that they did not contain any indication whatever of the fundamental point of the analysis, namely, that the total electrode loss is the sum of the heat conduction loss, due to the temperature difference at the two ends, plus *half* the Joulean heat. On the contrary, the minimum, which Mr. Hering then proposed to determine (page 301), was the minimum of the sum of the total conduction loss and the total Joulean heat loss, and he added, "if the total loss is really the sum of the two."

That, by making the current and therefore the Joulean heat large enough, an electrode can be forced to feed heat in both directions, was indeed brought out at that meeting, namely, by Mr. Hansen in his paper then presented and under discussion. I quote the following sentence from Mr. Hansen's paper (page 285). "The inference is, of course, that the electrode is at least as hot as the furnace interior by reason of the C^2R heating and not alone blocking the flow of heat from the furnace outward,

it actually feeds heat in both directions, the main portion naturally going to the cooling water jacket."

This experimentally-established fact of Mr. Hansen—that the Joulean heat can be raised to such an amount that it blocks the flow of heat from the furnace into the electrode—I have used as the foundation of the analysis in my paper.

For practical reasons, the establishment of the conditions under which the flow of heat from the furnace into the electrode is just blocked, seems a suitable starting point of analysis. If the Joulean heat furnishes what we may call the "rational" amount just required for this purpose, all the heat produced in the interior of the furnace will be usefully employed within; that is, as it should be. If the Joulean heat is smaller than this rational amount, heat will pass out of the furnace interior into the electrode, so that part of the heat produced within the furnace is wasted and lost to the outside. If, on the other hand, the Joulean heat is larger than the rational amount, then a maximum of temperature will be produced somewhere within the electrode; from this maximum point the temperature will decrease to both sides, and heat will flow off to both sides. The practical disadvantage is that on account of this temperature maximum within the electrode, not only the heat leakage from the electrode into the walls is increased, but the furnace lining or the walls in contact with the electrode at this point are liable to destruction. Hence, both an increase of the Joulean heat above the rational value and a decrease below it result in practical disadvantages.

Mr. Hering prefers as a starting point of the problem to find the condition of minimum electrode loss. Under certain simplifying assumptions this leads to the same results as our starting point, so that it is simply a matter of taste which starting point one will choose. But under conditions as they exist in practice, with heat leakage from the electrode, the two starting points lead to different results, as I will show later on.

Whatever the starting point which we may select, it is important to keep these two principles always in mind—first, to prevent an overheating of electrode and walls due to a temperature maximum within the electrode, and, secondly, to make the best possible use of the energy. As far as we can combine both principles we should try to do so.

As to the leakage of heat from the electrodes into the furnace walls, I agree with Mr. Hering that this represents the chief source of error in the formulas so far published. It will, therefore, not be out of place to give here briefly a very simple and rough extension of the analysis taking account of this heat leakage. As it is intended only as a rough first approximation, we will assume (as in Mr. Hering's analysis) mean values for the thermal and electric conductivities, which we treat as independent of the temperature within the temperature limits under consideration. In a similarly rough way we will assume a mean heat leakage coefficient L, defined as the amount of heat given off per second from one square centimeter of electrode surface to the adjacent furnace wall. L depends, of course, on the temperature difference between electrode and wall, therefore indirectly on the current, further on the material of the wall, on the nature of contact between electrode and wall, etc., but we will treat it for the present as constant, just like the conductivities.

In the same way, as in § 1 of my paper, we investigate again what happens in the infinitely small space between the cross-sections at the distances x and $x + dx$ from $x = 0$. The heat $-sc_h\, dT/dx$ passes per second into this space through the first cross-section. The heat $-sc_h\, dT/dx - sc_h\, dx\, d^2T/dx^2$ passes out of this space through the second cross-section. The heat $i^2 dx/(c_e s)$ is produced in this space from electrical energy. The heat $LC dx$ leaves the space on account of heat leakage into the surrounding wall (C being the circumference of the electrode cross-section).

The condition that the temperature does not change in time is, therefore,

$$\frac{d^2T}{dx^2} = \frac{LC}{sc_h} - \frac{i^2}{c_e c_h s^2}$$

By integration we get

$$T = T_0 - ax + bx^2$$

where

$$a = (T_0 - T_l)/l + LCl/(2c_h s) - i^2 l/(2c_e c_h s^2)$$

and

$$b = LC/(2c_h s) - i^2/(2c_e c_h s^2)$$

The heat which passes from the furnace interior into the hot electrode end ($x = 0$) is

$$(1) \qquad sc_h (T_0 - T_l)/l + \tfrac{1}{2} LCl - i^2 l/(2c_e s)$$

Now $sc_h (T_0 - T_l)/l$ is the heat which would be conducted away if there was no electric current passing; this term represents the simple heat conduction due to the difference of the temperatures T_0 and T_l.

$\frac{1}{2} LCl$ is one-half the total heat leakage into the walls.

$i^2 l/(2c_e s)$ is one-half the total Joulean heat produced in the electrode.

Hence we have the result that the heat which passes from the furnace interior into the hot electrode end equals the heat conducted away by simple heat conduction due to temperature difference between the two electrode ends, plus one-half the heat leakage to wall, minus one-half the Joulean heat. In this respect the heat leakage into the wall, therefore, has an effect like a decrease of the Joulean heat.

In the same way we find that the heat which passes from the cold electrode end into the water jacket equals the heat conducted away from the furnace by simple heat conduction due to temperature difference between the two electrode ends, minus one-half the heat leakage to wall, plus one-half the Joulean heat. We may express these results in the statement that the Joulean heat flows off impartially one-half into the furnace and one-half into the water jacket, while the heat, which leaks afterwards from the electrode into the furnace wall, is sucked into the electrode impartially one-half from the furnace and one-half from the water jacket.

But the heat given off from the cold electrode end into the water jacket no longer represents the total "electrode loss," since the latter should obviously include the heat lost from the electrode by leakage into the walls. At least, if the electrode is operated in a rational way, so that there is no maximum of temperature in its interior, then there will be a steady drop of temperature also in the furnace walls from the inside to the outside, and all the heat which leaks from the electrode into the walls will pass to the outside and be lost.

The total "electrode loss" will be, therefore,

$$(2) \qquad sc_h\,(T_o - T_l)/l + \tfrac{1}{2}\,LCl + i^2 l/(2c_e s)$$

It equals the heat conducted away by simple heat conduction, due to the temperature difference between the two electrode ends, plus one-half the Joulean heat, plus one-half the heat leakage from electrode to wall. (It may sound paradoxical to state that only one-half of the heat leakage from electrode to wall is lost, not the whole. The explanation is that, as we saw before, the heat which leaks from the electrode into the walls comes one-half from the furnace, and this half is lost, and the other half from the water jacket around the cold electrode end. This latter half, therefore, simply disappears in the water jacket around the cold electrode end and reappears in the atmosphere or in the water jacket around the walls. It evidently does not represent a loss from the inside of the furnace to the outside).

While the above results were derived from rough assumptions, which are nothing more than a first approximation, it is easy to prove in exactly the same way, as in § 3 in my paper, that the general rules concerning the superposition (combination by addition and subtraction) of the three heat quantities are valid in general. It is unnecessary to give this proof here, since we could copy the proof in § 3 almost word for word. The chief difference is that B — A is now the total Joulean heat generated within the electrode less the total heat leaking from the electrode into the furnace walls. y is one-half this value.

Of course, the specific formulas for temperature, etc., derived above, will not hold in general (except as far as they can be made to hold true by special artificial definitions invented particularly for this purpose).

Without entering into a detailed discussion of these results, we may point out two obvious facts:

First: The heat leakage into the walls increases with the length and circumference of cross-section (or, what is the same, with the lateral surface) of the electrode. It has nothing to do with the area of the cross-section. Therefore, if we take heat leakage into the walls into consideration, it is no longer true that the electrode loss depends only on the ratio of length to cross-section. It is, therefore, now meaningless to try to find that

value of this ratio for which the electrode loss would be a minimum. Of course, if, for instance, cross-section and circumference are given, we can find that value of the length for which the electrode loss is a minimum, etc.

Second: The condition of temperature equilibrium at the hot electrode end and the condition of minimum electrode loss are no longer the same condition, as a comparison of (1) and (2) will show. It is interesting to investigate what corrections are necessary to fulfil these two conditions, if we want to improve upon the formulas in which heat leakage is neglected.

The term of correction to be applied is simply $\frac{1}{2}$ LCl, but in (1) it has a sign opposite to that of the Joulean heat loss, while in (2) it has the same sign.

With respect to (1), the heat leakage into the wall has the same effect as though the Joulean heat or the current i was diminished. Hence, in order to obtain temperature equilibrium at the hot electrode end, we must make the electrode longer or smaller in cross-section than would follow from the simple formulas in which the heat leakage into the walls is neglected.

On the other hand, with respect to (2), the heat leakage into the wall has the same effect as though the Joulean heat or the current i was increased. Hence, in order to make the total electrode loss (2) a minimum, we must make the electrode shorter or larger in cross-section than would follow from the simple formulas in which heat leakage into the walls is neglected. If we do this, the remarkable result is obtained that we make the total electrode loss a minimum, although a positive amount of heat passes from the furnace interior into the hot electrode end. There is, therefore, no danger of overheating the electrode or the surrounding walls, since the temperature will drop steadily within the electrode from the inside to the outside.

However, it would be perfectly correct to argue that what we are really after, if we want to make the best use of the energy, is not minimum electrode loss, but maximum furnace efficiency. This leads again to another condition, but we will not dwell further here on this point, besides stating that to fulfill this condition, we must make the electrode longer (or smaller in cross-section) than would follow from the condition of minimum electrode loss. When the resistance of the electrodes is neg-

ligibly small compared with the resistance of the furnace charge, the conditions of maximum furnace efficiency and minimum electrode loss become identical.

To sum up our argument, if we base the calculation of the length of the electrode (cross-section, circumference, current and temperature being given) on the condition of temperature equilibrium at the hot electrode end, we get an upper limit for the length, above which we should not go, because we would then produce a temperature maximum somewhere within the electrode. Nor is it wise to select exactly this length, because any accidental rise of the current above its normal value during operation would cause the same trouble.

On the other hand, if we base the calculation of the length of the electrode on the condition of minimum electrode loss, we get a lower limit for the length, below which we should not go, because we would then only increase the loss without any compensating advantages. We should make the electrode length rather greater than this lower limit by a certain amount, in order to raise the efficiency of the furnace as a whole.

It is at least comforting to know that the value of length found from the simple formulæ in which the leakage of heat into the walls is neglected, lies between the upper and lower limits just discussed.

Because Mr. Hering's test for determining mean conductivities is based on an arrangement in which the heat insulation is as perfect as possible, and because in electric furnace practice the conditions are different, it is perfectly true to say that this test, if carried out according to Mr. Hering's original instructions, or in the way proposed at the end of my paper, will give values which are not directly applicable in practice. But the value of this test is due to the fact that we are working under well-defined conditions and know exactly what we are doing. We thus get well-defined values of the conductivities, or directly of l/s, and if we check them in the way suggested by me, we have a set of constants the precise meaning of which we know and on the accuracy of which we can rely, although we may not apply them directly (without correction) for electrode design.

As to the correction to be made for practical purposes, the general rules, which can be derived from theory, have been given

above. But the numerical value of the correction factor will depend largely on the conditions of each special case. What we need now are more thorough, systematic experimental investigations, such as those of Mr. Hansen. These investigations may suitably be carried out in two steps:

First, a determination of "mean conductivities" (Hering) or li/s (§ 3 of my paper) under experimental conditions in which heat leakage is excluded.

Second, experimental analysis of the different items, which make up the total electrode loss if heat leakage is present, on the basis of the general principles outlined above.

MEMOIR
ON THE METHODS OF TREATMENT OF SIMPLE AND COMPLEX ORES

OF

MOLYBDENUM, TUNGSTEN, URANIUM, VANADIUM

BY

GUSTAVE GIN

Part III.—Vanadium.

Translated by Jos. W. Richards and W. S. Landis.

This Memoir was awarded the Frenzel Prize by the American Electrochemical Society.

HISTORICAL.

Vanadium was discovered in 1801 by Del Rio, Professor of Mineralogy in the City of Mexico, in a lead mineral from Zimapan. In this brown mineral Del Rio noticed the presence of a metallic material very different from chromium and uranium, which he called Erithronium, on account of the red color of its salts.

This discovery was announced to the Institute of France by a letter from Humbolot and Bompland dated the 2d Messidor of the year IX, inserted in 1804 in the annals of the Museum of Natural History, and translated in Gilbert's Annalen.

Humbolot and Bompland sent a sample of the new mineral, which was analyzed by Collet-Descotils, chief engineer of the Bureau of Mines. This chemist after too short an investigation concluded that no new metal was present and that chromium was the only rare metal in the mineral.

Del Rio on his part likewise concluded that the supposed new

element was nothing more than chromium. Erithronium had, therefore, fallen into oblivion, when Sefstrom, Director of the School of Mines at Falun, Sweden, on examining a specimen of wrought iron which showed remarkable malleability and tenacity, recognized in it a very small proportion of a new element. The iron came from Taberg in Smaland, and Sefstrom concluded that the pig iron contained a rather large proportion of the unknown body, the greater part of which must have been lost in the refining slags. He succeeded later in isolating several centigrams of the oxide of vanadium from the refining slags, and recognized many of the properties of its salts.

Later, in 1830, he continued his researches in the laboratory of Berzelius, extracted from the Taberg refining slags a much larger quantity of the material and made an extended study of vanadium compounds. He named the element from Vanadis, the principal goddess of Scandinavian mythology. His researches were controlled and confirmed by Berzelius, who tried to isolate the element, but unsuccessfully. He tried to reduce the oxides of vanadium by carbon in a brasqued crucible, also by hydrogen, but obtained in both cases only a lower oxide having a nearly metallic luster. He believed that he had reduced them by potassium, obtaining thus a grayish powder which gave a metallic luster when burnished. Finally he used a method analogous to that which Rose devised for the isolation of titanium, heating chloride of vanadium in a current of dry ammonium gas. He thus obtained a material with brilliant metallic luster, which, however, was not yet pure vanadium.

Schafarick took up the same line and showed that Berzelius had obtained only the nitride of vanadium and not the metal, for the product disengaged ammonia when fused with caustic potash. Schafarick himself was deceived, believing that he had obtained metallic vanadium by passing chloride of vanadium and hydrogen through a strongly heated glass tube. The metallic looking material which was formed in this manner proved to be, however, di-vanadyl chloride.

Johnston and Crudnowicz repeated the experiments of Berzelius and shared his errors, classifying vanadium with tungsten and molybdenum.

Rammelsberg and Schabus fixed more exactly the classification

of vanadium in the system of elements by showing the resemblance of Vanadinite to Apatite and Pyromorphite.

Roscoe followed later and proved that his predecessors had only handled an inferior oxide, oxychloride or nitride, and had not known the pure metal. He succeeded in isolating the metal by reducing the tetrachloride by hydrogen.

Roscoe proved that vanadium formed an oxide, vanadyl, which plays the part of a radical like uranyl, and demonstrated that vanadium should be classed with phosphorus and arsenic. These observations were completed and confirmed by the work of St. Claire Deville, Ditte and Caron.

In 1894 Moissan made "cast" vanadium in the electric furnace.

In 1900 Gin obtained ferro-vanadium by the metallothermic process of Goldschmidt, by the reduction by aluminium; later by several electrolytic processes. Since that ferro-vanadium has been manufactured industrially by Paul Girod and by the Vanadium Alloys Company.

PREPARATION OF PURE VANADIUM.

The preparation of pure vanadium is a difficult operation by a purely chemical method. The reduction of vanadium oxides by carbon is possible only at the temperature of the electric furnace and furnishes not pure metal, but a carbide or carburized "cast" metal.

The reduction by hydrogen was attempted by Berzelius, Uhrlaub and Schafarick, but without results.

The reduction by potassium has never succeeded. With aluminium powder the reduction only takes place when in the presence of a metal capable of alloying with the vanadium.

Dry ammonium gas acting on the tri-chloride furnishes only a nitride. The reduction of this same compound by hydrogen is not complete. Sodium reduces vanadium chloride, in an iron tube, giving a metal containing 91.5 percent vanadium.

Outside of the electrolytic processes, which will be described later, only one process has produced pure vanadium, that devised in 1869 by Roscoe, a description of which is as follows:

CHEMICAL PREPARATION OF VANADIUM.

Reduction of the Tetra-Chloride by Hydrogen.

This method of preparation as carried out by Roscoe requires delicate manipulation. The principal difficulty is in the great oxidizability of vanadium at a high temperature. It is, therefore, absolutely necessary to use the tetra chloride free from oxy-

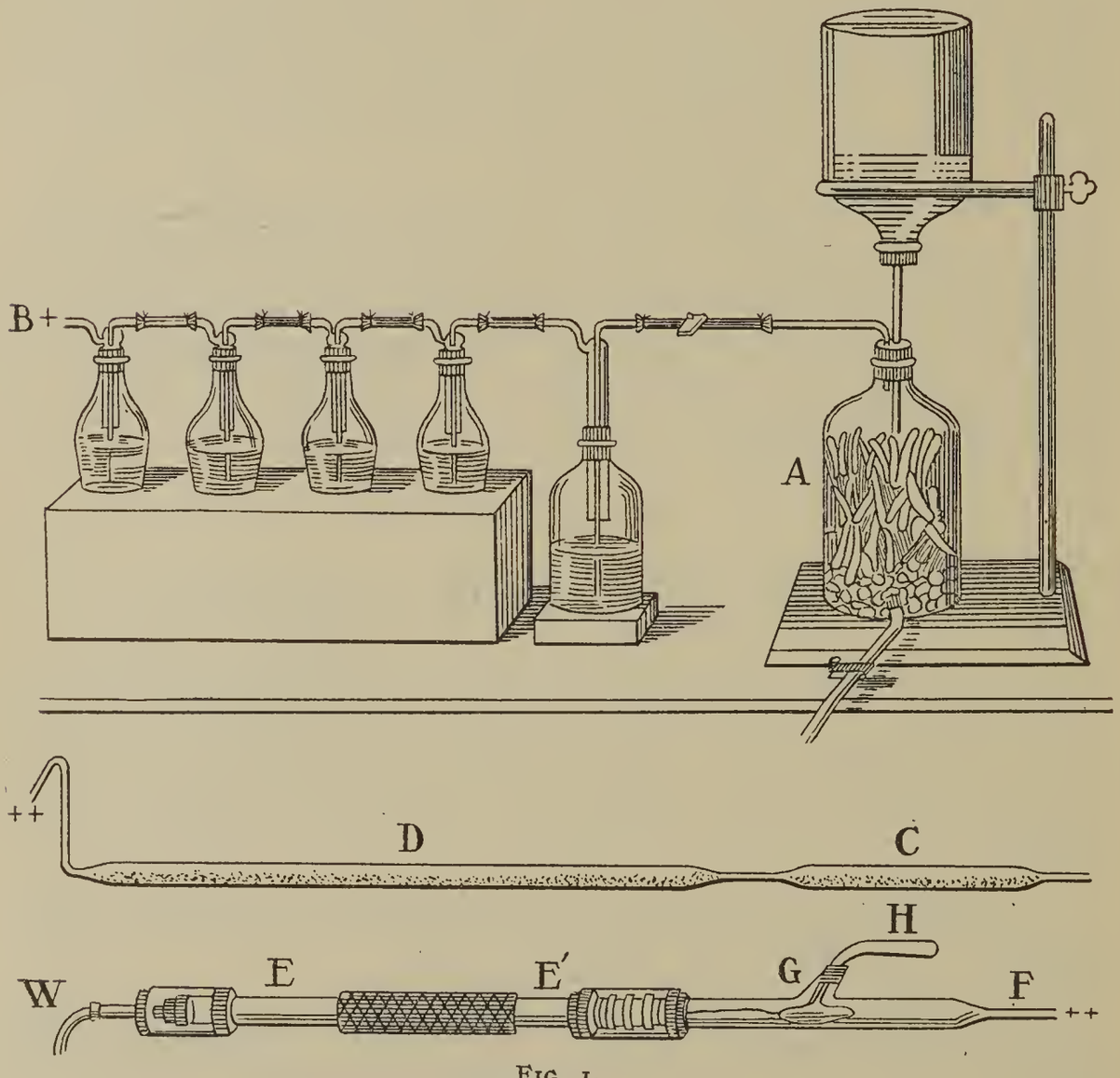


FIG. 1.

chloride, and to avoid the presence of even the minutest quantity of oxygen in the current of hydrogen gas, which must remain uninterrupted during 40 to 80 hours. Finally, vanadium attacks vessels of glass and earthenware.

After many attempts, Roscoe adopted the arrangement shown in Fig. 1.

The arrangement used consists of apparatus for producing

hydrogen, A, which can work continuously for a week by the addition of acid in the upper flask and the removal of the sulphate of zinc formed from the lower flask. The hydrogen gas passes through a washing flask containing acetate of lead, a second containing nitrate of silver and, finally, a third set of three containing sulphuric acid which has been previously boiled. To remove any oxygen which might be mixed with the gas the current of gas after leaving the washing flasks is carried through a tube CD, the first part of which contains platinum black, which is heated during the passage of the hydrogen and the last part D containing phosphorus pentoxide, kept in place by wads of cotton. All the joints are made with rubber fastened to the tubes with copper wire and covered over with paraffin. A tube bent at right angles connects the tube CD to the other part of the apparatus. This portion consists of the tube EE of porcelain placed in a Hofmann furnace and protected around the central part, which is strongly heated, by a short iron tube. This porcelain tube is connected to the hydrogen tube by means of the glass connector FF'. The joint between the two tubes is made by rubber carefully tied and this joint protected by a glass cylinder; the space between the joint and the glass cylinder is filled with mercury or melted paraffin. A similar joint is made at the other end of the porcelain tube.

To introduce the bichloride of vanadium into the apparatus without exposing it to the air, the tubulure G is employed, in which is fixed the bent tube H, containing the bichloride of vanadium sealed in with hydrogen gas. A platinum boat intended to receive the bichloride is placed as indicated in the figure, immediately below the bent tube.

The experiment is commenced by passing hydrogen 12 hours through the apparatus, to dry it and remove air. The curved tube is taken away, filled and replaced rapidly at G so that the crystals of bichloride are in the horizontal part. The tube is then revolved in the ground joint G so that the bichloride falls into the platinum boat. This boat is then drawn into the porcelain tube by means of the platinum wire, the end of which passes through the rubber at the other end of the apparatus. When the boat is in its place the platinum wire is cut off at the end of the glass tube and another tube dipping into sulphuric acid is

attached to this first one. The ground stopper of the tubulure is then carefully closed with paraffin, and hydrogen is passed through the apparatus six hours.

The porcelain tube is then heated up to bright redness by the Hofmann furnace and kept at this temperature the whole period of reduction. Hydrochloric acid commences to form rapidly, and the operation is continued until the hydrogen leaving the apparatus contains no more hydrochloric acid.

The time of reduction varies from 40 to 80 hours according to the quantity of the material used, which varied in Roscoe's experiments from 1 to 4 grams.

Physical Properties.

The vanadium obtained by Roscoe was a brilliant grayish powder, strongly reflecting light and as white as silver under the microscope. It was not magnetic and could not be agglomerated by compression. Density at 15°, 5.5. When melted together it had a metallic fracture, was white like silver and possessed marked prismatic crystallization. The melting point was above 2,000°, and it was considered by Moissan as one of the very refractory metals.

The spectrum of vanadium has been studied by Thalen, Hasselberg, Lockyer and Baxandall.

The specific heat as found by Matignon and Bonnet equals 0.1233 at 0°.

Chemical Properties.

Vanadium is not attacked by dry air at ordinary temperatures. At a high temperature it burns in oxygen with bright incandescence. Heated in air, it oxidizes first to a brown dioxide, then to a black trioxide, and finally to a blue tetraoxide, which is finally transformed to vanadic acid, V_2O_5. In moist air at ordinary temperatures it coats itself with a brown powdery coating.

It is attacked by chlorine gas with incandescence at temperatures above 100°. It fixes nitrogen at high temperatures forming a nitride. Hydrochloric acid does not attack it hot or cold. Sulphuric acid when concentrated does not attack it cold, but when hot forms a greenish yellow solution. It dissolves easily in nitric or hydrofluoric acids; with nitric acid there is a disengagement

of brown vapors and there results a blue solution. Fused alkalies are decomposed by vanadium forming alkali vanadates. Vanadium when hot attacks glass and porcelain, forming a black silicide of vanadium, even causing the fracture of the vessels.

It alloys easily with platinum, making a brittle alloy. It is insoluble in molten silver. It is soluble in melted pure iron, but insoluble in liquid cast iron. It forms alloys with aluminium, tungsten, molybdenum, iron, nickel and chromium, which alloys will be studied more in detail later.

Chemical Relations of Vanadium—Atomic Weight.

The place vanadium holds in the table of elements has been largely discussed. For a long time it was considered a member of the tungsten and molybdenum group, but the researches of Roscoe in 1867 proved that it should be placed with phosphorus and arsenic, which it resembles closely in its pentoxide, and in the vanadates, which are isomorphous with phosphates and arsenates. Thus Vanadinite, a chloro-vanadate of lead $3Pb_3V_2O_8.PbCl_2$ is isomorphous with Pyromorphite, a chloro-phosphate of lead, $3Pb_3P_2O_8.PbCl_2$ and Mimetite, a chloro-arsenate of lead, $3Pb_3As_2O_8.PbCl_2$.

Berzelius determined the atomic weight of vanadium as 68.5, but he evidently weighed vanadyl, and not the metal.

Kenngott and Struve, not being able to explain the isomorphism with the arsenates and phosphates, supposed that vanadinite contained an acid with less oxygen than vanadic acid.

Struve, guessing at the facts, proposed to change the atomic weight in such manner as to give vanadic acid the formula V_2O_5. Baumgarten arrived at a similar conclusion by his work in replacing the phosphorus in fluo-phosphate of sodium by vanadium, and obtained crystals isomorphous with the original salt. Roscoe settled the question completely by showing that the supposed vanadium of Berzelius was an oxide (vanadyl) and that the compositions of the oxides of vanadium were represented by the formulas V_2O_2, V_2O_3, V_2O_4 and V_2O_5.

Roscoe, estimating the chlorine in the compound $VOCl_2$, found 51.25 for the atomic weight of vanadium. The International Committee on Atomic Weights has adopted 51.27.

MINERALS OF VANADIUM.

Formation of Vanadium Minerals.

The formation of vanadium minerals in the crust of the earth is an interesting geological question, which has been clarified by the memoir on this subject by M. Ditte, presented to the Academy of Sciences, Paris, May 30, 1904, and to which those interested in this geological side are referred.

Oxides.

Vanadine, V_2O_5, the only natural oxide known, is a yellow earthy coating found on the native copper from Lake Superior.

Sulphides.

Sulvanite, $3Cu_2S.V_2S_5$, was found by Goyder in rather important quantities near Burra, Australia.

Patronite, discovered in the neighborhood of Cerro de Pasco, Peru, by Riza de Patron, is a dark green material resembling olivenite, with a fibrous fracture. It has a dark green streak, hardness 3.5, specific gravity 2.65. The analysis by Mr. Handy, of Pittsburgh, gave:

SiO_2	10.88%	V	16.08%
Al_2O_3	3.45	S	54.06
Fe	3.45	Mo	0.50

Vandates.

There is a considerable number of mineralogical species of vanadates, the most important of which is Vanadinite, more properly speaking a chloro-vanadate of lead.

Vanadinite, $3PbV_2O_8.PbCl_2$, is a reddish-orange or yellow to red mineral, vitreous in luster, crystallizing in hexagonal prisms, and isomorphous with Pyromorphite and Apatite, the chlorophosphates of lead and lime, respectively. Its density varies from 6.8 to 7.2, and it is very brittle.

It was first found at Zimapan, Mexico, then at Charcas, San Luis Potosi, Mexico, in the mines of argentiferous lead. At Charcas it is found in regular veins 5-6 millimeters thick and of a waxy green color.

It has been found likewise in Carinthia, at Mount Obir, near Berezowsk in the Urals, at Wanlookhead, Scotland, in Cape

Colony and the Transvaal. It has been found recently in Cordoba, Argentine Republic, in rather important quantities. Several mines in Arizona and the silver mines of Marqueza in Chili also contain it. St. Seine noticed it in the copper mines near Coquimbo, Chili, and at Taleuna in the same province.

In Spain, in the Concession of Baxeres, in the province of Seville, it has been worked by a French company at Santa Marta, and also by the "New Vanadium Alloys Co."

The following are some analyses of Vanadinite:

	San Luis Potosi	Spain			Cordoba		Arizona	Rammelsberg
	Lhote	Gin	Gin	Gin	Gin	Gin	Gin	
V_2O_5	14.42	18.24	15.80	16.38	18.64	13.70	19.12	19.12
$PbCl_2$	. . .	7.56	7.82	8.20	9.20	6.24	8.90	9.14
PbO	43.08	64.20	57.10	51.40	66.90	51.16	68.10	77.28
CuO	10.01	2.90	1.74	2.50	0.44	1.86	0.28	. . .
ZnO	0.26	. . .	0.24	0.90	. . .	0.30	. . .	. . .
CaO	5.48	0.50	3.40	1.92	1.06	4.80	0.40	. . .
SiO_2	13.20	3.46	8.24	15.38	1.40	20.00	0.56	. . .
Al_2O_3		0.26	0.43	0.26	. . .	0.20	0.10	. . .
P_2O_5	. . .	0.14	0.29	0.16	0.84	0.90	1.24	1.41
Loss and not determined	8.44	2.74	4.94	2.90	1.52	1.84	1.30	. . .

Vanadinite without chlorine is a different mineral, having the name of *Descloisite,* which always contains some zinc and manganese. It crystallizes in the orthombic system in small black or greenish brown crystals; density 5.8-6.1. It is found in the Argentine Republic near Cordoba, and in New Mexico and Spain.

The following are some analyses of the mineral:

	Le Plata		Spain
	Damour	Rammelsberg	Gin
V_2O_5	22.46	22.74	25.40
PbO	54.70	56.48	52.80
CuO	0.90	....	1.38
ZnO	2.04	16.60	3.20
MnO	5.32	1.16	3.44
MnO_2	6.	....	....
Fe_2O_3	1.50	....	....
SiO_2	3.44	....	10.65
H_2O	2.20	....	1.48
Loss and not estimated	1.44	3.02	1.65

The Vanadinite of Carinthia and *Psittacinite* are species allied to Descloizite and not very well defined. The mineral *Dechenite* may be likewise classed with Descloizite, as a variety rich in zinc. It is found in the Rhine Pfalz at Niederschlittenbach as an orange powder in mammilary masses; it has likewise been found near Dahn in the Rhine Palatinate.

Eusynchite, $PbO.3ZnO.2V_2O_5$, in the form of a crusty, concretionary earth, is similar to Dechenite and has been found at Hofsgrund in Baden. It is reddish yellow in color and has a density of 5.27-5.53.

Analyses of Dechenite:

Analyst:	Dana	Kobell	St. Chamond	Gin
Origin:	Hofsgrund	Dahn	Spain	Bavaria
V_2O_5	24.22	34.92	15.80	23.12
PbO	57.88	48.76	50.35	56.80
CuO	0.63	.....	6.06	1.44
ZnO	15.80	16.38	12.80	12.60
MnO	.....	.....	.....	0.80
SiO_2	.....	.....	2.15	1.10
P_2O_5	1.44	.....	.....	0.46
As_2O_5	0.50	.....	0.86	0.62
H_2O	2.34	.....	.....	2.
Loss	.....	.....	.....	1.06

Analyses of Eusynchite:

	Czudnowiez		Rammelsberg
V_2O_5	23.546	19.164	24.22
PbO	56.469	53.911	57.66
ZnO	16.782	21.414	15.80
SiO_2	3.203	5.5511	.....

Areoxene is a vanado-arsenate of lead and zinc, in dark red crystals, density 5.3, found in Bavaria and in Dahn in the Rhine Palatinate.

Brackebuschite is a variety similar to the preceding, but contains more iron and manganese.

Cuprodescloizite is the name given to the above minerals when they contain more than 6 percent copper.

Chileite contains a still higher proportion of copper. It is a reddish brown mineral with fibrous fracture.

Analyses of Cuprodescloizite and Chileite:

	Cuprodescloizite		Chileite
	Nicolardot	Pisani	Domeyko
V_2O_5 ..... 22.47	18.95	17.40	13.33
As_2O_5 ..... 0.28	3.82	4.78	4.68
P_2O_5 ..... 0.17	0.18	.....	0.68
$PbCl_2$	.....	.....	0.37
PbO 54.03	54.93	53.90	51.97
ZnO 12.62	12.24	11.40	.....
CuO 8.13	6.74	8.80	16.97
MnO	.....	3.20	.....
FeO	0.06	.....	.....
SiO_2	.....	.....	1.33
CaO	.....	.....	0.58
Fe_2O_3, Al_2O_3	.....	.....	3.42
H_2O 2.52	2.74	.....	.....
Loss and not determined	.....	.....	6.67

Volborthite, a hydrated vanadate of copper, may be regarded as the extreme of this transformation; it often contains lime and crystallizes in little hexagonal tables, color olive-green to yellow, pearly luster, density 5.55. It is found in the Urals and between Minsk and Ekaterinenbourg in Siberia where it was discovered by Hess.

Calcovolborthite contains up to 12 percent lime and is regarded as a separate species. It is found in the neighborhood of Friedrichshohe, Thuringerwald.

Analyses of Volborthite and Calcovolborthite:

	Volborthite		Calcovolborthite		
	Gin	Genth	Credner		
			Green	Light Green	Gray
V_2O_5 ..... 38.80		14.55	36.58	36.91	39.02
P_2O_5 ..... 0.58		.....	.....	.....	.....
CuO 40.90		40.70	45.15	38.90	37.27
CaO 8.40		4.80	12.28	17.40	16.55
MnO		.....	0.40	0.53	0.52
BaO 2.20		.....	.....	.....	.....
MgO 1.40		1.52	0.50	0.87	0.92
SiO_2 ..... 6.70		.....	0.40	0.77	0.76
H_2O 4.60		.....	4.62	4.62	5.05
Loss, etc.... 2.42		.....	.....	.....	.....

Pulcherite is a vanadate of bismuth from Schneeberg, containing 27 percent V_2O_5 and 73 percent Bi_2O_3..

Dewalquite or *Ardennite* is a vanado-silicate of aluminium and magnesium found at Ottrez, Belgium.

Eosite is a vanado-molybdate of lead.

Roscoelite is a mica near to Lepidolite and rich in vanadium; density between 2.9 and 3. It is found in porphyry in the Granite mine near Coloma, California, near to Red Gorge and Sutter's Mill, where gold was first discovered.

Analysis of Roscoelite:

	Black		Black
V_2O_5	20.56%	K_2O	7.59%
SiO_2	47.69	Na_2O	0.19
Al_2O_3	14.10	Li_2O	trace
Fe_2O_3	1.67	H_2O	4.96
MgO	2.00		

Carnotite, studied in 1899 by Friedel on specimens discovered in 1895 by Chas. Poulot in Montrose County, Col., is a yellow pulverulent mineral which shows traces of crystallization, though mixed with quartz sand. It has likewise been found in Brazil and Chili. It corresponds to the formula

$$K_2O.2UO_3.V_2O_5.3H_2O.$$

Analyses of Carnotite:

	Friedel	Gin	Gin
V_2O_5	20.12	18.02	11.02
P_2O_5		0.08	0.16
UO_3	63.54	56.90	35.10
K_2O	10.37	8.60	5.70
H_2O	5.90	5.20	3.32
SiO_2		8.94	41.18
CaO		2.06	3.08
Loss and not determined		0.20	0.44

Finally, many minerals which do not properly belong to the vanadium minerals, such as certain clays, bauxites and ashes of many soft coals and anthracites of South America, particularly those of the anthracites of Yauli, Peru, may be included as sources of the metal.

Yauli is a small town situated 180 kilometers from Lima at an

altitude of 4,200 meters, and on the railway between Lima and Aroya. The climate is very rigorous and vegetation is lacking. Several silver and copper mines are exploited in the neighborhood. Eight kilometers from Yauli is Lake Llacsacocha, near which are the deposits of anthracite and the mines of Austria, Margarita, San Bonito, Progreso, Clemencia et Chola.

The Austria mine is the only one worked. A syndicate called the Paris-Yauli was formed to exploit these anthracite mines. In 1895 it transferred its rights to the Société Anonyme de Yauli, which opened the Austria mine. The work was greatly hindered by the climate and transportation, and resulted disastrously. Also the furnaces used were closed and expensive to build; they have been replaced by a simple structure of four walls with a grate between them on which the anthracite is burned. No attempt is made to utilize the heat of its combustion. The ashes, after collecting, were loaded on llamas and carried to the port of Calloa.

Financially, the results were not equal to the expenditures, and the company soon ceased operation. In 1903 a French-Italian syndicate tried to regenerate the enterprise, but without success.

In spite of this, it appears that working in this manner ought to give profitable results, if carried out with sufficient working capital. The anthracites of Yauli contain at least 12 percent of ash, which itself contains at least 25 percent vanadium, or 38 percent vanadic acid, representing more than 40 kilograms of V_2O_5 in a ton of anthracite. It appears that V_2O_5 could be made on the spot, utilizing the heat of combustion of the anthracite for performing the chemical reactions required.

Mr. Mourlot gives the following analyses of the ash of the Yauli anthracites:

Soluble in acids		Insoluble in acids	
V_2O_5	38.5	SiO_2	13.6
SO_3	12.1	Al_2O_3	5.5
P_2O_5	0.8	Fe_2O_3	9.4
Fe_2O_3	4.1	MgO	0.9
Al_2O_3	4.		29.4
CaO	8.44		
K_2O	1.8		
	69.74		

Vanadium likewise exists in a large number of iron minerals;

those of Taberg contain 0.12 percent V_2O_5, and of the Harz 0.2 percent.

Pitchblende contains up to 3 percent vanadium.

Sphalerite from Tunis contains likewise a sensible amount. Finally, there are certain poorly characterized minerals containing vanadium, such as certain greenish earths, found near Denver, Colorado, which contain 2-5 percent vanadic acid.

To sum up, vanadium is widely distributed in nature, but rich vanadium minerals are rare. However, since attention has been so closely called to it discoveries of vanadium minerals have succeeded each other rapidly, and minerals which had previously been supposed non-vanadiferous are now recognized as possible sources of vanadium.

PRINCIPLES OF THE TREATMENT OF VANADIUM MINERALS.

Vanadium minerals are generally treated for V_2O_4 or V_2O_5. Three methods are employed:

A. Chlorination.

The mineral is calcined in the presence of reducing gases so as to remove arsenic and transform V_2O_5 into V_2O_4. A current of dry chlorine gas is then passed over the mass, forming the volatile oxy-trichloride; on passing this into water it decomposes, forming hydrated vanadic acid. This manner of operating is rather a laboratory method than an industrial process. Gin has nevertheless, put it into practice, using ferric chloride as a source of chlorine.

B. Solution in Acid.

The vanadium may be dissolved in nitric acid, hydrochloric acid or sulphuric acid and the base metals precipitated out. The solution is then precipitated as meta-vanadate of ammonium, insoluble in chloride of ammonium. The meta-vanadate is calcined for V_2O_5.

C. Transformation into Alkaline Vanadate.

The material is fused with alkali carbonate and nitrate. The

alkaline vanadate is dissolved from the insoluble material and the impurities in solution removed by precipitation with suitable reagents.

We will examine the various applications of methods based on the above general reactions, commencing with a description of processes which are now only historical, such as the treatment of the slags of Taberg and of Creusot, the earths of Cheshire, and the anthracite ashes of Yauli.

METHOD OF SEFSTROM FOR TREATING THE REFINING SLAGS OF TABERG.

The slags, finely ground, were treated in a reverberatory furnace, mixed with carbonate and nitrate of soda in equal proportions. After prolonged calcination the mass was cooled, pulverized and washed with boiling water. The filtrate, neutralized by nitric acid, was treated with a soluble salt of lead or barium, forming a precipitate of vanadate of lead or barium containing also the phosphoric acid, silica, zinc and aluminium present. The precipitate was treated with concentrated sulphuric acid, giving a red solution of vanadium sulphate, $V_2O_5.2SO_3$. Alcohol was then added to reduce to vanadous sulphate, $V_2O_4.2SO_3$, forming a blue solution. After filtration hydrofluoric acid was added to eliminate silica as gaseous silicon fluoride. After this operation it was evaporated to dryness to remove sulphuric acid, leaving a residue containing V_2O_5 and some phosphoric acid, zirconia and alumina. This residue was fused with saltpeter, forming vanadate of potassium, while the alumina and zirconia remained insoluble. The material being dissolved in water and filtered, ammonium chloride in excess was added to the filtrate to precipitate the vanadium as meta-vanadate of ammonium, which is insoluble in excess of ammonium chloride. The phosphate remains in solution. The ammonium meta-vanadate on being calcined leaves pure V_2O_5.

METHOD OF NORBLAD FOR THE TREATMENT OF THE SLAGS OF TABERG.

Norblad attacked the slags in fine powder by concentrated sulphuric acid. On saturating the excess of acid with metallic iron,

evaporating to dryness, taking up with water and evaporating, the ferrous sulphate can be crystallized out. The mother liquor was decanted, evaporated to dryness and the residue calcined.

The calcined mass, of a yellow color, was fused with potassium nitrate and taken up with water. After separating out the insoluble ferric oxide, evaporating to separate out the excess of nitre by crystallization, the remaining solution was saturated with carbon dioxide to precipitate the silica, which was filtered out. The vanadic acid was precipitated as meta-vanadate of ammonium.

WITZ AND OSMOND'S METHOD FOR TREATMENT OF THE CREUSOT SLAGS.

The slags from the basic Bessemer converters at Creusot, which used the cast iron obtained by the reduction of oölitic ore from Mazenay, contained over 1 percent of vanadium.

The pulverized slags were digested with cold hydrochloric acid until the latter was saturated, and the solution was then evaporated to 36-37° B. The solution thus obtained, containing the phosphate of the tetroxide of vanadium in hydrochloric solution, was filtered to separate silica, and used directly in certain operations in dyeing.

To extract the vanadic acid from it, it was neutralized by the addition of the proper proportion of alkaline acetate, which precipitated the tetraoxide of vanadium, a bluish gray material. This, on being filtered out and roasted at low redness, was again dissolved in ammonia to an orange solution as the ortha-vanadate, and was transformed into the meta-vanadate by boiling. The meta-vanadate was precipitated by ammonium chloride in excess. For many years this process of Osmond and Witz enabled the works at Creusot to manufacture the whole of the vanadium salts employed in dyeing, their annual product representing 60,000 kilos of vanadium.

Afterwards, vanadium salts having been displaced in the dyeing industry, Schneider & Company abandoned the treatment of the slags. The renewal of this industry has been rendered impossible by the discovery of comparatively rich vanadium minerals in later years.

ROSCOE'S METHOD FOR THE TREATMENT OF THE CHESHIRE OCHRES.

These materials are found in the Lower Triassic beds near Alderley Edge, in Cheshire, and consist of cupriferous gray earths in which Roscoe discovered vanadium in 1868. At that time the earths were treated for copper, cobalt and nickel, and the residue from this treatment was a blue liquor containing 2 percent vanadium. Roscoe undertook the extraction of vanadium from this and succeeded. Later Roscoe extracted vanadic acid from the earth itself in the following manner: The pulverized earth was attacked by hydrochloric acid, forming a solution of chlor-hydrate of vanadium tetroxide. This was treated with milk of lime and bleaching powder. The precipitate contained the vanadium, lead, iron, arsenic and phosphoric acid. It was calcined, mixed with carbon and heated to remove arsenic. The calcined mass was pulverized and mixed with carbonate of soda and heated until all the vanadium was transformed into soluble vanadate. After treatment with water, the solution was acidified with hydrochloric acid and treated with a current of sulphur dioxide in excess to reduce the arsenic to arsenious acid, the latter then being precipitated by a current of hydrogen sulphide gas.

The blue solution remaining was exactly neutralized with ammonia, the precipitated vanadium hydrate filtered off, washed, exposed to the air for partial oxidation, finally oxidized by nitric acid, and then calcined to crude vanadic acid. This was purified by digestion with ammonium carbonate solution, taking up the vanadium and leaving behind the iron, alumina, and sulphate of calcium. After filtration, the meta-vanadate was precipitated by ammonium chloride in excess and purified by repeated crystallization. The meta-vanadate was roasted to vanadic acid, which was then re-dissolved in ammonia so as to separate silica and phosphoric acid.

The Alderly Edge Copper Mining Company have abandoned the exploitation of this material for copper, and transferred their operations to the cupriferous earth of Mottram Saint Andrews, where carbonate and arsenate of copper are found. They treated also at the same time the cupro-vanadiferous material of Harm Hill near Shrewsbury. In 1871 they showed to the British Asso-

ciation at Liverpool samples of ammonium vanadate, which commanded at that time $350 per kilogram. The mine at Shrewsbury was later worked by the Magnesium Metal Company of Patricroft, and the price of vanadate of ammonium was rapidly reduced to $11 per kilogram.

TREATMENT OF BAUXITES AND ARGENTIFEROUS CLAYS.

St. Claire Deville treated bauxites with hydrochloric acid to dissolve the lime. The residue was fused with soda, taken up in water, treated with hydrogen sulphide to precipitate silica and alumina, the sulpho-vanadate remaining in solution as alkaline sulpho-vanadate. The addition of acetic acid to this solution precipitated vanadium sulphide, which was then roasted to vanadic acid.

Beauvallet treated the vanadiferous clays of Gentilly with a boiling solution of sodium carbonate. The solution was neutralized by sulphuric acid, ammonia added and then sulphide of ammonium. Alumina and silica being precipitated, the solution of ammonium sulpho-vanadate was precipitated as insoluble sulphide which was converted into vanadic acid.

These two last named processes have not been employed industrially.

TREATMENT OF THE VANADIFEROUS ASHES OF YAULI.

Mr. Helouis devised the method of treating the ashes obtained from the combustion of the anthracite of Yauli. These ashes contain 18-40 percent of vanadic acid. They are first digested with a concentrated solution of caustic soda. After prolonged treatment water is added, the liquid brought to boiling and filtered hot on asbestos cloth. The residue left on the filter is again treated with fresh alkali solution in a similar manner and the two solutions after filtration united. These solutions contain almost all the vanadium, and part of the silica, alumina and phosphoric acid of the original material. Next neutralize carefully with dilute nitric acid, evaporate to dryness and take up in water, the silica remaining insoluble. The filtered liquid is exactly neutralized with ammonia and the alumina precipitated. On again filtering the vanadium is precipitated as ammonium meta-vanadate by an excess of ammonium chloride crystals. The

precipitate is washed with a cold saturated solution of ammonium chloride, compressed and dried at 110°. It is then washed once with cold water to eliminate sodium chloride. The washing is terminated with water slightly acidulated with nitric acid. The material is then dried and calcined to V_2O_5 in porcelain dishes.

This process of Mr. Helouis was not used long. We have seen that the company at Yauli fell into difficulties, a victim of the financial combinations which existed at its organization.

TREATMENT OF VANADINITE.

Wohler's Process: Wohler treated vanadinite with nitric acid, which dissolved the vanadium and other metals and left behind the insoluble silicious gangue. The acid solution was neutralized by ammonia in slight excess, treated with a current of hydrogen sulphide to precipitate lead, copper, etc.; the red solution was filtered and dilute sulphuric acid added to it, precipitating vanadium sulphide. Vanadic acid was obtained from this by an oxidizing roast.

Herrenschmidt Process: Herrenschmidt has described this process himself in the Comptes Rendus, October 24, 1904, as follows:

The mineral treated is lead vanadate from Santa Marta (Spain), containing 12-14 percent V_2O_5 and 50 percent Pb. It is fused in a reverberatory furnace with sodium carbonate and carbon, forming argentiferous lead and a slag containing vanadate, silicate and aluminate of soda, and ferric oxide. This slag is only slightly soluble in water; it is melted in a reverberatory furnace and, while molten, air is blown through it, which completely oxidizes the vanadium to pentoxide. The molten mass is then run into boiling water to granulate it and washed; after three washings the residue, consisting principally of alumina, silica and iron oxide, contains less than 2 percent vanadic acid. Per ton of mineral treated there is obtained 280 kilograms of this residue, and a solution containing 95 percent of the original vanadium. This residue may be further treated by solution by sulphuric acid.

The solution of sodium vanadate does not contain alumina. To remove silica in it a part is evaporated to the consistency of

syrup and concentrated sulphuric acid, 66° B., is added, forming sulphate of soda and precipitating vanadic acid, but all the vanadium is not precipitated, about 10 percent remaining in solution. The mixture is then brought into contact with the rest of the impure solution, and after energetic stirring the solution is passed through a filter press. All the silica in the silicate of soda is precipitated, while the vanadium remains in solution.

The solution of alkaline vanadate, free from silica and other impurities, is concentrated and the vanadium precipitated by excess of sulphuric acid. On evaporating to dryness to drive off the sulphuric acid, and washing, there is obtained V_2O_5, 92-95 percent pure.

The above is Hammerschmidt's own statement of the process, which contains no really novel points and, besides, does not eliminate arsenic or phosphoric acid.

Herrenschmidt has also proposed to precipitate vanadate of sodium by means of sulphates of iron, nickel or copper, thus obtaining directly double oxides which may be used in metallothermic reactions for the production of ferro-vanadium, nickel-vanadium and cupro-vanadium.

The French patent, No. 328421, of January 10, 1903, to Mr. Herrenschmidt gives a resumé of this process as follows:

"A process consisting in the reduction of the vanadate of lead ores by carbonate or sulphide of sodium, soda or the sulphate of sodium, and carbon; or the treatment of Colorado earths by attacking with sulphuric acid of 50° B., and precipitating the vanadium as sodium vanadate by sulphate of iron, or chloride of iron, with or without the addition of caustic soda or carbonate of soda in a liquid of sirupy consistency. This operation takes place cold. The oxides obtained may be reduced in such manner to obtain alloys of iron, copper or other metals, according as the precipitation has been made by one or the other of these metals.

To obtain vanadium itself, the liquid containing sulphate of iron and the vanadium is treated by an oxidizing agent, chlorine or bleaching powder, or soda to precipitate the iron as sesquioxide, while the vanadium goes over to the state of sodium vanadate.

A NEW METHOD OF TREATING VANADINITE.

The author (Gin) has elaborated a method of treating vanadinite consisting in its preliminary transformation into alkaline vanadate by means of alkaline carbonates, in the dry way, thus obtaining a soluble vanadate which is separated from the impurities. This method permits of first obtaining the base metals, then obtaining the vanadic acid free from silica, alumina and phosphoric acid.

Assume a vanadiferous mineral at hand of the percentage composition and formula (the latter represented in kilogram-molecules per ton of ore) as follows:

Percentage Composition.		*Formula of Mineral.*
V_2O_5	9.13%	$0.5\,V_2O_5 + 2SiO_2 + 0.1Al_2O_3 + 0.2P_2O_5 + 2.4PbO + 0.6CuO + 0.8CaO + 0.2Fe_2O_3 + 0.8CO_2$.
SiO_2	12.00	
Al_2O_3	1.02	
P_2O_5	2.84	
PbO	53.52	
CuO	4.76	
CaO	4.48	
Fe_2O_3	3.20	
CO_2	3.52	
Undetermined	5.53	

The exceptional amount of phosphoric acid present is due to apatite in the ore.

First Operation—Partial Electric Reduction.

The ore is treated in the electric furnace with a limited amount of carbon, liberating the base metals and producing a slag containing the vanadium in the form of the tri-oxide.

This operation may be symbolized by the following chemical reaction, expressed per ton of mineral used:

$$\text{1000 Kg ore}\left\{\begin{array}{l}0.5V_2O_5 + 2SiO_2 + 0.1Al_2O_3 + 0.2P_2O_5 + \\ 2.4PbO + 0.6CuO + 0.8CaO + 0.2Fe_2O_3 + \\ 0.8CO_2\end{array}\right.$$

$$\text{60 Kg carbon} + 5C =$$

Products obtained:

Base metals: $2.4Pb + 0.6Cu$.

Vanadiferous slag: $0.5V_2O_3 + 2SiO_2 + 0.1Al_2O_3 + 0.2P_2O_5 + 0.8CaO + 0.4FeO$ (307.5 Kg).

Carbon monoxide disengaged: $5.8CO$.

This first reduction yields about 490 kilograms of lead and 35 kilograms of copper, which may be separated from each other by known methods. Because of the moderate amount of carbon used, a small amount of these metals reamins in the slag.

There is obtained about 300 kilograms of slag containing:

V_2O_3	24.0%	P_2O_5	9.3%
SiO_2	39.0	CaO	15.0
Al_2O_3	3.3	FeO	9.4

This slag is quite fusible, so that no flux need be added to it.

Second Operation:—Oxidation of the slag to alkaline vanadate.

The slag may be treated with nitric acid to dissolve V_2O_3, from which V_2O_5 is obtained by passing through meta-vanadate of ammonium, but this method is inadvisable because the nitric acid also dissolves the calcium phosphate, which precipitates with the ammonium meta-vanadate, making it impure. Likewise reduction of the tri-oxide is never complete in the presence of phosphoric acid. It is preferable to treat the slag in the following manner:

The slag is finely ground, and heated gently in a current of air to oxidize the V_2O_3. The dark green powder changes to an indigo blue color, and the slag becomes:

$$0.5V_2O_4 + 2SiO_2 + 0.1Al_2O_3 + 0.2P_2O_5 + 0.8CaO + 0.2Fe_2O_3$$

Sodium carbonate and a little nitrate is then added to complete the oxidation of the V_2O_4 to V_2O_5, and the temperature is raised until the mass fuses. There is thus formed sodium meta-vanadate, along with silicate, aluminate and phosphate. The mass is cooled in the air and then crushed fine to be treated in the wet way.

Third Operation:—Solution and precipitation of the V_2O_5.

The crushed material is treated with boiling water to dissolve the meta-vanadate, silicate, aluminate and phosphate of sodium,

while the small quantities of lead and copper present remain dissolved with the iron.

The solution is cooled and a current of carbon dioxide passed through it to precipitate silica and alumina, leaving a mixed solution of carbonate, phosphate and vanadate of soda. This reaction is carried on cold because the precipitation of silica by CO_2 is a reversible one. In a boiling solution, silica displaces carbon dioxide. Into the filtered solution a calculated amount of ammonium carbonate is added and this concentrated until the vanadium precipitates as meta-vanadate, while the sodium phosphate remains in solution:

$$Na_2V_2O_6 + (NH_4)_2CO_3 = (NH_4)_2\ V_2O_6 + Na_2CO_3$$

This precipitation is favored by the presence of ammonium chloride, which is added as crystals to the solution.

The precipitate is washed with pure water, dried in the air, then in an oven, then washed with dilute nitric acid to remove the last traces of alkaline phosphate or carbonate.

If the precipitate contains considerable quantities of silica and phosphoric acid it should be roasted and re-dissolved in ammonia. After filtration, the addition of ammonium chloride crystals will precipitate ammonium meta-vandanate, which is filtered, dried and calcined to V_2O_5, which may be obtained as a powder or melted down into slabs. During calcination about 70 percent of the combined ammonia may be regained.

Treatment of the Wash Waters.

Burnt lime is put into the preceding solution from the ammonium vanadate and heated. The ammonium in excess in solution distills off and is regained. The lime precipitates phosphoric and carbonic acids, leaving in solution caustic soda, which is afterwards carbonated by the gas from the lime kilns.

PRACTICAL DATA.

I. *Materials Consumed:*	Per ton of Ore	Per ton of V_2O_5
Vanadinite ore, 9.1% V_2O_5	1,000 kg.	11,400 kg.
Coke for reduction	70 "	800 "
Electrodes	35 "	400 "
Carbonates of soda (lost)	22 "	300 "
Nitrate of soda	44 "	600 "
Carbonate of ammonium	14 "	160 "
Coal for crystallization, evaporation, calcination, etc.	175 "	2,000 "

	Per ton of Ore	Per ton of V_2O_5
II. *Net Products:*		
Vanadiferous slag, 25% V_2O_5	308 kg.	3,500 kg.
Lead	473 "	5,400 "
Copper	37 "	420 "
III. *Electrical energy consumed*	600 kw-h.	6,840 w-h.

Net Cost per Ton of Vanadic Acid in January, 1909.

I. *Materials Consumed:*		
Vanadinite ore, 9% V_2O_5, 11,400 kg @ $80 per ton	$1,112.00	
Coke for reduction, 800 kg @ $8 per ton	6.40	
Electrodes, 400 kg @ $7 per 100 kg	28.00	
Sodium carbonate, 300 kg @ $4 per 100 kg	12.00	
Sodium nitrate, 600 kg @ $8 per 100 kg	48.00	
Ammonium carbonate, 160 kg @ $12 per 100 kg	19.20	
Coal, 2,000 kg @ $6 per ton	12.00	
Refractory materials and miscellaneous	12.40	
		$1,250.00
II. *Electrical energy,* 6,840 kw-hours @ $0.01		$ 68.40
III. *Labor*		9.60
IV. *Repairs*		8.00
V. *Sinking fund*		16.00
VI. *Packing*		4.00
VII. *Interest on material and product in stock*		10.00
VIII. *General expenses, rebates, transportation*		24.00
Total		$1,390.00

From this total must be deducted the value of the base metals obtained.

Variations of the Preceding Process.

The use of the electric furnace may not be convenient in most localities. The first electric operation may be replaced by water jacket furnace smelting, thus separating directly lead and copper. The slag is granulated, and after cooling, charged into the hearth of a reverberatory furnace fired by producer gas. At 800° the flame is made strongly oxidizing, and sodium carbonate and nitrate is added to ensure quiet fusion and oxidize the vanadium tetroxide, but not the silica and alumina. The following reactions take place:

Slag—$0.5V_2O_5 + 2SiO_2 + 0.1Al_2O_3 + 0.2P_2O_5 + 0.8CaO + 0.2Fe_2O_3$.

Carbonate of Sodium—$3.4Na_2CO_3$.

The products obtained are:

$$0.5Na_2V_2O_6 + 2Na_2SiO_3 + 0.1(3Na_2O.Al_2O_3) + 0.4Na_3PO_4 + 0.8CaO + 0.2Fe_2O_3 + 3.4CO_2.$$

The new slag is cooled, powdered very finely and digested with boiling water, leaving behind silicate of lime and oxide of iron. The solution is decanted, cooled by CO_2, filtered, and the vanadium precipitated by ammonium carbonate, as previously described.

Second Variation of the Process.

The process as described was worked out for ores containing 15-17 percent V_2O_5 and 3-4 percent silica. Later, on treating materials containing 9-10 percent V_2O_5 and 10-15 percent silica it was found that it was inadvisable to use so much soda because of the loss of alkali salts in the process, and it was found preferable in such cases to use sodium bisulphate. The vanadiferous slag was then treated with sodium bisulphate, as will be explained further on.

In this case the use of the mixed method results in doing away with ⅔ to ½ of the weight of the mineral for electric reduction, and the treatment by bisulphate reduced by ½ the weight of material under treatment.

THE TREATMENT OF VANADINITE BY ALKALINE BISULPHATES.

The author (Gin) has devised a method of utilizing bisulphates in attacking vanadinite, salts which are easily and cheaply obtained. The fusion takes place in a furnace in a pan of wrought iron or special cast iron, such as are used in the manufacture of sodium sulphate from common salt and sulphuric acid.

The bisulphate is first melted with closed doors, and then the ore is added, finely pulverized, little by little. The decomposition of the carbonates present causes lively effervescence; the heating must be done gently, so as not to decompose the bisulphate. When the reaction is finished, the melt is tapped out into moulds of silicious iron or 15 percent ferro-silicon. The product is yellow at first, and rapidly absorbs moisture from the air, turning green, and finally bleaches. It contains sulphates of lead and sodium, and sulphate of divanadyl and vanadic oxide. There is first formed vanadic sulphate by the reaction:

$$3Pb_3V_2O_8,PbCl_2 + 32KHSO_4 = 16K_2SO_4 + 3(V_2O_5.2SO_3) + 10\,PbSO_4 + 2HCl + 15H_2O.$$

This sulphate is partially reduced by the SO_2 present. The material is crushed between cast iron rolls and digested with water in a vessel heated by steam and through which air is blown. The liquid is decanted and sent through tanks, in which the residual mud containing silica and sulphates of lead and calcium is washed out. The vanadium in it may then be precipitated by ammonium chloride in acid solution, the phosphoric acid remaining in solution. If the ore contains copper in considerable quantities, the operation may be varied as follows:

The copper, silver and arsenic may be precipitated by hydrogen sulphide; on adding ammonia and then passing hydrogen sulphide again, the iron and alumina are precipitated and eliminated by filtration, the vanadium remaining in solution as sulpho-vanadate; the vanadium may be precipitated by careful neutralization with dilute hydrochloric or sulphuric acid.

To economize in ammonia the free acid in solution may be removed by the addition of sodium sulphide, which sets free H_2S, precipitating the silver and copper.

In fact, it is preferable to suppress entirely the use of ammonia and to add sufficient sodium sulphide to precipitate the base metals and transform at once the sulphate of vanadic acid into sodium-hypo-sulpho-vanadate. After filtering and washing the precipitate it is transformed into vanadium sulphide by hydrochloric acid.

The excess of hydrogen sulphide disengaged during the above operation may be caught and used to produce from sodium carbonate the sodium sulphide necessary.

Perret patented in France, December 7, 1904, No. 348633, the process of treating vanadium ores consisting in attacking them by fused alkaline bisulphates, calcining, catching the SO_3 evolved, taking up the residue in boiling water, filtering and treating finally with ammonium chloride, precipitating ammonium meta-vanadate which is calcined to V_2O_5. This patent of Mr. Perret was applied for several years after the publication of the method of the author.

Cost of Vanadic Acid by the Alkaline Bisulphate Method.

Assume a vanadinite of the following composition corresponding to the subjoined formula in kilogram molecules per ton.

V_2O_5	16.42%	
PbO	51.29	
$PbCl_2$	8.34	
CuO	2.40	*Formula (per ton).*
ZnO	0.81	$0.9V_2O_5 + 2.3PbO + 0.3CuO$
CaO	2.24	$+ 0.1ZnO + 0.3PbCl_2$
SiO_2	15.—	$+ 0.4CaO + 2.5SiO_2 +$
Al_2O_3	0.20	$0.02Al_2O_3 + 0.01P_2O_5$
P_2O_5	0.14	
Loss	3.16	

On treating with sodium bisulphate in quantity 50 percent more than that theoretically required there is obtained:

Soluble: $0.9V_2O_2(SO_4)_3 + 0.3CuSO_4 + 0.1ZnSO_4 + 0.3CaCl_2 + 0.02Al_2(SO_4)_3\ 0.01P_2O_5 + 5.86Na_2SO_4 +$ excess $NaHSO_4$.

Insoluble: $0.1CaSO_4 + 2.6PbSO_4 + 2.5SiO_2$.

Treating the solution with $\frac{n + 5.86}{2}$ molecules of sulphide of sodium, where n is the number of molecules of bisulphate used, precipitating the base metals, there is needed 2.7 molecules of hydrochloric acid to precipitate the vanadium sulphide.

If the number of molecules of bisulphate used is 9, one can calculate that the treatment of a ton of ore requires practically:

Sodium bisulphate	1080 Kg.
Sulphide of Sodium (anhyd.)	580 "
Hydrochlorid acid, 20° B.	315 "

from which we obtain about 155 Kg of V_2O_5.

Cost of Treatment per Ton of Vanadic Acid.

I. *Raw Materials and Reagents:*

Ore, 16% V_2O_5, 6,500 kg @ $9 per unit per ton	$ 950.40
Sodium bisulphate, 14,000 kg @ $8 per ton	112.00
Sodium sulphide, 6,100 kg @ $20 per ton	122.00
Hydrochloric acid, 20° B., 2,100 kg @ $12 per ton	25.20
Miscellaneous	10.40
	$1,220.00

	Amount forward	$1,220.00
II.	*Coal for drying, concentration and calcination,* 4,000 kg @ $6 per ton	$ 24.00
III.	*Labor*	9.00
IV.	*Maintenance of furnaces*	8.00
V.	*Sinking fund*	15.00
VI.	*Packing*	4.00
VII.	*Interest on ore and products in stock*	10.00
VIII.	*General expenses, rebates, transportation*	24.00
	Total	$1,314.00

The treatment costs about $0.26 per kilogram of V_2O_5. There is required 6,500 kilograms of ore to produce a ton of V_2O_5 from which is extracted 3,200 kilograms of lead and 120 kilograms of copper.

If the value of these metals amounts to $200, the manufacture of the V_2O_5 may be considered as costing nothing, provided the lead and copper are not charged in with the ore.

Variations of the Above Process.

Since a large part of the V_2O_5 is used industrially for the manufacture of ferro-vanadium by the metallo-thermic process, it is often advantageous to precipitate a mixture of oxides of vanadium and iron, which can be done as follows:

After attacking with bisulphate and taking up with boiling water, turnings and chips of iron may be added to the solution, which will reduce the vanadic acid present to vanadium tetroxide. This reduced liquor is decanted and the solution of 25 percent soda is run into it, which precipitates the vanadium and iron together as a dark gelatinous mass. This is first boiled for several hours, then separated on a filter press and washed. The clear liquor is tested for vanadium by a few drops of hydrogen peroxide, which turns a red color if any vanadium is still in solution. The filter press cakes are washed with pure cold water until the filtrate shows no precipitation with barium chloride. They are then dried in an oven and pulverized before packing in barrels. It is thus possible to make, according to the proportions of bisulphate and iron used, mixtures of 20-75 percent V_2O_5 and V_2O_4.

Captain Nicolardot states that this product contains a little phosphoric acid, which passes into the ferro-vanadium as phosphorus. The method is therefore to be proscribed when the ores are phosphoric. One company furnished ferro-vanadium con-

taining 0.44 percent phosphorus, which was refused by the consignees and which had to be returned to the makers.

The characteristic reactions of the process are:

I. Attack by sodium bisulphate

$$V_2O_5 + nSiO_2 + 6NaHSO_4 =$$

insoluble: $nSiO_2$

soluble: $V_2O_5.3SO_3 + 3Na_2SO_4$

II. Reduction by iron

$$V_2O_5.3SO_3.4H_2O + 3Na_2SO_4 + Fe + H_2O = V_2O_4.2SO_3.4H_2O + FeSO_4.H_2O + 3Na_2SO_4$$

III. Precipitation by soda

$$V_2O_4.2SO_3.4H_2O + FeSO_4.H_2O + 3Na_2SO_4 + 6NaOH =$$

soluble: $6Na_2SO_4$

insoluble: $V_2O_4.7H_2O + FeO.H_2O$

GIN PROCESS, USING FERRIC CHLORIDE.

At about 300° ferric chloride reacts on V_2O_5, giving the trichloride of vanadyl, which distils at 126°, and when caught in water decomposes, re-forming V_2O_5. This process is applicable to vanadiferous sands as well as to vanadinite. To apply it, a mixture of ferric sulphate and calcium or magnesium chloride with a vanadiferous mineral is charged into a muffle furnace having an iron condenser attached. Greenish yellow vapors of vanadyl trichloride pass over when the temperature is moderated. When the V_2O_5 has been all removed the vapors become reddish because of the appearance of ferric chloride, and the reaction is then stopped.

The reactions are as follows:

$$Fe_2(SO_4)_3 + 3CaCl_2 = 3CaSO_4 + 2FeCl_3$$
$$2FeCl_3 + V_2O_5 = 2VOCl_3 + Fe_2O_3.$$

A clear yellow fuming liquid is recovered in the condenser, which contains crystals of double chloride of iron and vanadyl. The liquid part is decanted and there is added to it slowly about ⅓ its weight of water, precipitating V_2O_5 according to the reaction:

$$2VOCl_3 + 3H_2O = V_2O_5 + 6HCl$$

When the precipitation is almost complete it is filtered through

asbestos, washed, dried and calcined. If the filtered clear liquid still contains vanadium, it is concentrated by boiling, which removes the larger part of the hydrochloric acid, which is caught. The V_2O_5 is then precipitated from the solution by milk of lime, washed, dried and calcined.

The crystals of double chloride of iron and vanadyl may be dissolved in water and treated by soda, which makes soluble sodium vanadate and precipitates iron. If it is desired, the ferro-vanadium compound may be returned the cycle of operations, as also the hydrochloric acid saved, and thus produce a truly cyclic process.

Application of the Above Process to Uranium-bearing Vanadium Ores.

The above described ferric chloride process is likewise applicable to minerals containing uranium, such as carnotite and uraniferous vanadiferous sands. The ore is mixed with ferric sulphate and magnesium or calcium chloride in quantities sufficient for the following reaction:

$V_2O_5.2UO_3.K_2O.3H_2O + 4FeCl_3 =$
$2(VO)Cl_3$—distilling at 126°.
$2(UO_2)Cl_2 + 2KCl$—soluble.
$2Fe_2O_3$—insoluble.

The vanadium is obtained as previously described by condensing the volatil chloride, followed by decomposition in water. The uranium remains in the residue as uranyl chloride or the double chloride of uranyl and potassium, both of which are very easily fusible. The residue is taken up by hot water, giving a yellow solution, which is filtered from the insoluble residue, and from which milk of lime precipitates hydrated uranium oxide.

Application of the Above Process to Vanadates.

This process is likewise applicable to the treatment of metallic vanadates, particularly Vanadinite. The reactions are as follows:

$$\underbrace{3Pb_3V_2O_8}_{\text{Vanadinite}} + PbCl_2 + 6Fe_2(SO_4)_3 + 18CaCl_2$$

$= 18CaSO_4 + 10PbCl_2 + 6Fe_2O_3$—precipitated.
$+ 6VOCl_3$—volatile.

The succeeding operations are as were described. This simple process supplants for vanadiferous ores most of the supplementary operations which are necessary to separate impurities from the solutions or the precipitates containing vanadium.

PROCESS OF THE VANADIUM ALLOYS COMPANY, COLORADO, FOR TREATING VANADIFEROUS EARTHS.

In Colorado, on Big Bear Creek, west of Telluride, is a vein of vanadiferous earth of Jurassic formation. There are two layers of white earth separated by black limestone. The vanadium occurs as granules or impregnations of Roscoelite, varying between greenish yellow and dark olive green. The ore contains 2-5 percent V_2O_3 and a small proportion of V_2O_5. Pure grains of this Roscoelite, carefully separated, showed on analysis:

V_2O_5	15-16% (V_2O_3)
SiO_2	45-46
Al_2O_3	16-19
Fe_2O_3	12-13
K_2O	6-8
H_2O	2.5-3.5

This ore is treated in the works at Newmire. The ore is broken up in jaw crushers and then pulverized to 20 mesh in rolls. This pulverized material is given an oxidizing roast with common salt (chloridizing roast). The mass is stirred until tests show all the vanadium in the state of V_2O_5. Steam is then introduced into the furnace, disengaging chlorine and hydrochloric acid. Taking as an example the ore as analyzed above, the reactions will be as follows:

$$(V_2O_3.9SiO_2.Fe_2O_3.2Al_2O_3.K_2O.2H_2O) + 2O = (V_2O_5.9SiO_2.Fe_2O_3.2Al_2O_3.K_2O) + 2H_2O.$$

$$(V_2O_5.9SiO_2.Fe_2O_3.2Al_2O_3.K_2O) + 2NaCl + H_2O = (V_2O_5.Na_2O) + 9SiO_2.Fe_2O_3.2Al_2O_3.K_2O) + 2HCl.$$

After roasting, the mineral is cooled, sprinkled with water, and taken to tanks for methodic leaching. The concentrated solutions are filtered and taken to precipitating vats where ferric sul-

phate is added, resulting in the precipitating of vanadate of iron containing 40-70 percent V_2O_5. The Newmire works treat 12 tons of ore daily.

MISCELLANEOUS PROCESSES FOR TREATING VANADIUM ORES.

J. H. Haynes and W. D. Engle have patented in America the process of treating the pulverized ores with a solution of an alkaline carbonate until the vanadium is dissolved. Decanting the solution, it is precipitated by slaked lime as calcium vanadate. This process has been known for a long time.

Bouffort patented in France, August 20, 1905, No. 357397, a process for treating ores of vanadium and vanadiferous slags consisting in obtaining soluble compounds of vanadium by the use of sodium peroxide at a low temperature. This reagent is expensive to use.

De Lucie patented in the United States a process of treating ores containing vanadium sulphide. Such ores are roasted and treated with sodium hyposulphite. If the vanadium in the roasted ore is as a lower oxide it passes to pentoxide when combining with soda. On washing one obtains a solution of sodium vanadate, which is precipitated by hypochlorite of calcium, regenerating the sodium hypochlorite. The precipitate of calcium vanadate is filtered and the solution used over for treating a new charge.

The author (Gin) has devised the following method of treating rich sulphide ores:

The ore is mixed with proper proportions of sodium sulphate and carbon and heated in a muffle furnace. Sodium sulphovanadate is formed according to the reaction:

$$V_2S_5 + 2Na_2SO_4 + 2C = Na_4S_5V_2O_2 + 2SO_2 + 2CO.$$

Washing the mass with boiling water dissolves the sulphovanadate to a red solution, which oxidizes rapidly and disengages hydrogen sulphide. Sodium vanadate in solution is formed under the influence of the hydrogen sulphide evolved and colors the liquor blue. Air is then blown through the liquor to oxidize it, and nitric acid is added. Precipitation is finally done by milk

of lime. The caustic soda solution resulting, when separated from the precipitate, may be converted into sulphide by treatment with hydrogen sulphide and returned to the first part of the operation.

TREATMENT OF THE CARNOTITE EARTHS OF COLORADO.

Ohly has given a description of the method of treating the uranium-bearing vanadiferous ores found in Colorado and Utah. They are disseminated through an earth which carries 0.5 to 0.75 percent V_2O_5 and 2 to 3 percent UO_3. Vanadium is extracted as follows:

The earth is pulverized, sieved through a 60-mesh sieve, and treated with a mixture of hydrochloric, sulphuric and nitric acids in earthenware vessels heated by a leaden steam coil. Acid is added until the liquid assumes a reddish color, and the heat is continued until the residue shows under the microscope no more yellow grains of Carnotite. The solution is decanted, it containing all the vanadium and uranium of the ore; a calculated quantity of chloride of ammonium is then added and the solution is evaporated to ⅓ its original volume. After cooling and standing over night, the clear liquid is decanted from the crystals of ammonium meta-vanadate. The latter are then redissolved and recrystallized, from a solution of chloride of ammonium, and washed with alcohol. The mother liquor is almost entirely free from vanadium and contains all the uranium. The process is based on the two following reactions:

$$V_2O_5 + 2HCl = 2VO_2Cl + H_2O.$$
$$VO_2Cl + H_2O + NH_4Cl = NH_4VO_3 + 2HCl$$

The meta-vanadate is then calcined until brown, the following decomposition taking place:

$$2NH_4VO_3 = V_2O_5 + 2NH_3 + H_2O.$$

V_2O_5 when heated to 440° melts to a brown mass which crystallizes on cooling.

The mother liquors contain sodium uranate. They are boiled for a half hour with excess of sodium carbonate, precipitating

the impurities. The filtrate is treated with caustic soda to precipitate the uranium. The reactions are as follows:

$$UO_3 + 3HCl = UO_2Cl_2 + H_2O + Cl_2.$$
$$2UO_2Cl_2 + 3Na_2CO_3 = Na_2U_2O_7 + 3CO_2 + 4NaCl.$$
$$Na_2U_2O_7 + 2NaOH = 2Na_2UO_4 + H_2O.$$

It is possible that in the second reaction intermediate double carbonates may be formed. The process corresponds almost entirely to the Patera method of analyzing pitchblende.

The sodium uranate may be sold as such or transformed into the green oxide U_3O_8.

ELECTROTHERMIC PREPARATION OF VANADIUM.

MANUFACTURE OF VANADIUM CARBIDE.

In the manufacture of vanadium steels the use of carburized vanadium introduces difficulties not only because of the proportion of carbon in the carbide or in the cast vanadium, but particularly because if the proportion of vanadium introduced into the steels is 1 percent, it would entail the simultaneous addition of 0.22 percent carbon if the carbide VC were used. It is evident that in treating mild steels only pure vanadium must be used.

Besides, properly speaking, vanadium carbide is not used in metallurgy, but the double carbide of vanadium and iron, in which the vanadium is supposed to exist in the form V_3C_2, or a compound containing a proportionately smaller amount of carbon. Gin has made in the electric furnace a ferro-vanadium corresponding to the formula:

$$V_3C_2 + Fe_3C + Fe_3$$

that contains 60-65 percent iron, 27-30 percent vanadium, and less than 7 percent carbon.

Such a ferro-vanadium would introduce into the steel a proportion of carbon about equal to that furnished by pure vanadium carbide; but, on the other hand, ferro-vanadium less rich in carbon than the above may be easily produced, using as raw materials a mixture of vanadium tri-oxide and carbon briquettes, and employing a furnace in which the material does not come in

contact with a carbon hearth or carbon electrodes. When the charge of oxide is being reduced, a certain quantity of vanadic acid may be thrown on the surface of the bath, and an arc used while this oxide removes carbon. It is thus possible to

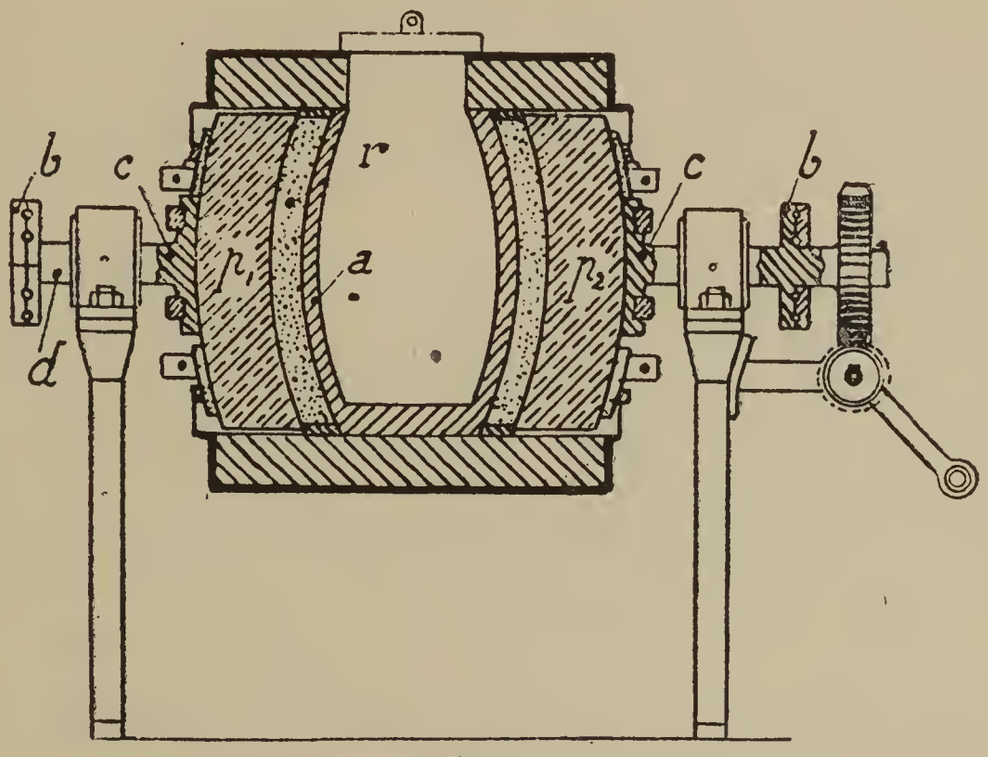


FIG. 2.

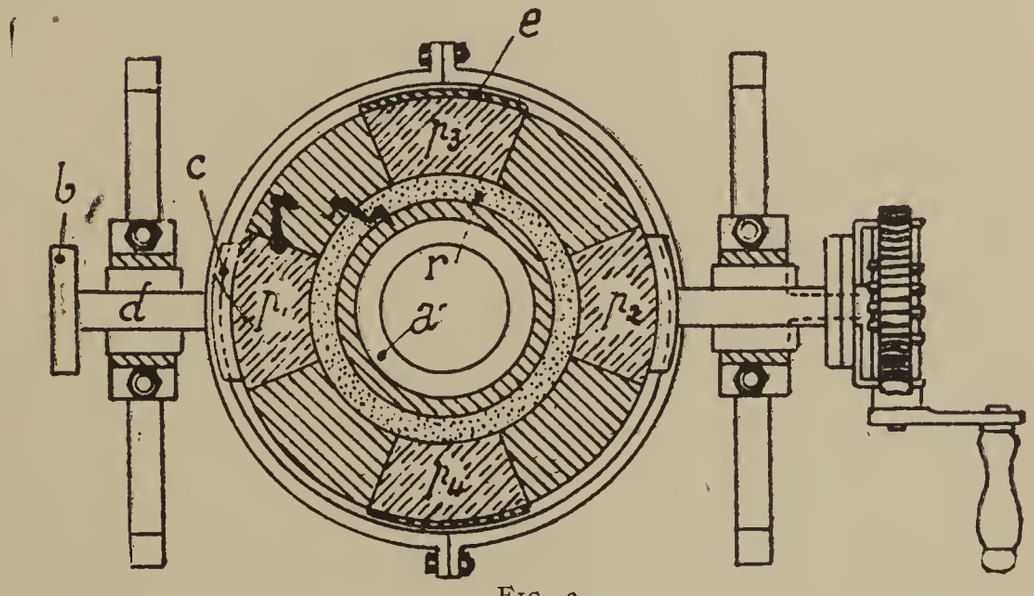


FIG. 3.

manufacture ferro-vanadium with 30 percent vanadium and only 1-2 percent carbon.

In the incorporation of vanadium carbide in steels it must be noted that the carbide has a high melting point and a very limited solubility in the steel. If the carbide constituent is allowed to

solidify first, its low density brings it to the surface and prevents it diffusing equally throughout the metal. When vanadium carbide is used it is necessary to pour the steel very hot and to cool quickly, so as to avoid the effects of segregation.

Manufacture of Vanadium Carbide by the Girod Process.

Girod manufactures at Albertville[1] ferro-vanadium, in his electric furnace, of which Captain Nicolardot gives the following information:

The materials to be treated are placed in a crucible or graphite or Veitsch magnesite. This is heated externally by electrical means, namely, by the principle of resistance constituted by means of powdered graphite mixed with metallic powder (iron chips) and mineral powder (silica, magnesia). The first is added to increase the conductibility, and the second to reduce it. By properly mixing these two, a resistor of any desired resistance may be made. The current passes through this resistor from the positive poles p_1 and p_2 and out through the negative poles p_3 and p_4, supported by refractory bricks. A horizontal axis D permits rotating the furnace even during the passage of the current. Two plates b b fastened to the axle take the current, and two plates c c of bronze conduct it to the poles p_1 and p_2. Iron bands hold these plates in position. Between the bands and the poles are insulating layers of asbestos. The top and bottom of the furnace are formed of sheet iron covered with a mixture of magnesia and silica.

To facilitate the starting, the poles p_1 and p_2 and p_3 and p_4 are connected by thin iron wires, which become red hot and thus heat the graphite around them; or a high tension of 70 to 80 volts may be used to start the furnace, which is afterwards reduced to 20-25 volts when running. The operation of this furnace is very simple and the obtainable temperature very high. It is possible to melt in it ferro-vanadium and ferro-tungsten carrying 80 percent tungsten. The temperature of the furnace may be easily maintained constant.

Giron manufactures ferro-vanadium in an electric furnace similar to the one used in the manufacture of steel. He obtains

[1] Revue d'Artillerie, March, 1904.

easily ferro-vanadium with 30, 40 or even 50 percent vanadium and carbon below 4 percent. This carbon is obtained by refining the carburized metal with vanadic acid.

MANUFACTURE OF VANADIUM SILICIDE.

Gin has obtained this silicide, V_2Si, by reducing in the electric furnace a mixture of V_2O_5, silica and coke, in the proportions shown by the reaction:

$$3V_2O_5 + SiO_2 + 13C = 2V_2O_2 + V_2Si + 13CO.$$

This corresponds to 10 parts vanadic acid, 1 part silica and 3 parts coke. A current density of 200 watts per square centimeter section of the electrodes is suitable. Part of the V_2O_5 is volatilized in the operation, the loss being diminished, however, if trioxide is used instead of vanadic acid, according to the reaction:

$$3V_2O_3 + SiO_2 + 7C = 2V_2O_2 + V_2Si + 7CO$$

Using commercial materials, Gin obtained a silicide of the following percentage composition:

Vanadium	71.16
Iron	5.40
Silicon	22.16
Carbon	0.22
Loss and not determined	0.56

The manufacture of the double silicide of iron and vanadium is still more easy than that of vanadium silicide and is obtained by reducing V_2O_3 by means of high ferro-silicon. Gin has manufactured it in the following manner: Sixty percent ferro-silicon is pulverized to a fine powder; this is intimately mixed with V_2O_3; the mixture is agglomerated with 4-6 percent tar and moulded into briquettes of ovoid form or bullets; this is charged into a furnace with a stamped magnesia hearth. The reduction takes place according to the following reaction:

$$4V_2O_3 + 4FeSi_3 + 7C = (4FeSi_2 + 3V_2Si) + (SiO_2 + V_2O_3) + 7CO.$$

The silicide obtained had the following percentage composition:

Vanadium	45.04
Iron	33.52
Silicon	19.40
Carbon	0.62
Loss and not estimated	1.42

Gin has likewise manufactured vanadium silicide and the double silicide of iron and vanadium by reducing the tri-oxide of vanadium in the electric furnace by carborundum, and by a mixture of carborundum and high ferro-silicon:

$$4V_2O_4 + 4SiC = V_2Si + 3SiO_2 + 3V_2O_2 + 4CO.$$

$$3V_2O_4 + 2SiC + 4FeSi_2 = (2Fe_2Si + V_2Si) + (7SiO_2 + 2V_2O_2) + 2CO.$$

PREPARATION OF FERRO-VANADIUM LOW IN CARBON.

Preparation from Carburized Vanadium.

The carburized cast vanadium, or ferro-vanadium, is placed in a Gin induction furnace, and vanadic acid thrown in on the surface of the bath. The vanadic acid is reduced to a lower oxide, but not to the metallic state. The proportion of carbon in the alloy may be reduced in this manner at least 1 percent. The alloy is then cast into ingot moulds of magnesia or bauxite, which have been previously heated. A similar refining may likewise be done with a mixture of vanadic acid and ferric oxide, but in this case vanadium always passes into the refining slag.

Preparation from Silicide of Iron and Vanadium.

The vanadium silicide may be refined in the same furnace, in which case a mixture of vanadic acid and lime, or meta-vanadate of calcium, is thrown on the surface. The silicon oxidized combines with the lime to form a fusible silicate. The refining reaction may be stated as follows:

$$3(2Fe_2Si + V_2Si) + 6V_2O_5 + 9CaO = 9CaSiO_3 + 6Fe_2V + 6V_2O_2.$$

The refining in the electric furnace with arcs playing on the surface of the bath is always difficult, as the force of the arc

scatters the slag, refining does not take place on the outskirts of the arc, and the temperature is so high that volatilization of the oxides takes place. In the region around the walls of the furnace the slag freezes and exerts no oxidizing action. It is therefore preferable to do the refining in an induction furnace with automatic circulation. The oxidizing materials are thrown on the surface of the bath and the refining reactions are very rapid because the circulation of the bath constantly renews the surfaces of contact.

The Electrometallurgical Company of Niagara Falls, in the United States Patents Nos. 858325, 858327, 858328, 858329, 866421, 866561, 866562, has claimed the manufacture of vanadium by treating vanadium oxides in the electric furnace by carbon silicide and iron, or carbon silicide and carbon, or by ferro-silicon. These patents appeared three years after the experiments of Gin on the same subject.

According to the author's experiments, ferro-vanadium very low in carbon may be made as follows: In an electric furnace with two movable electrodes, a layer of rich ferro-silicon is melted on the hearth, on the surface of which is thrown a mixture of vanadium tri-oxide and carbon to produce the reaction:

$$V_2O_3 + 2FeSi + 3C = V_2Si + Fe_2Si + 3CO.$$

The double silicide thus obtained is easily fusible in the electric furnace and may be run into a Gin induction furnace, where vanadium-tetra-fluoride is thrown upon its surface. Silicon fluoride is formed, which volatilizes and may be caught in a condensation chamber or in water as fluo-silicic acid. The reaction is:

$$V_2Si + Fe_2Si + 2V_2F_4 = Fe_2V_6 + 2SiF_4.$$

Vanadium oxy-fluoride, or lead fluoride, may likewise be used for the removal of the silicon according to the reactions:

$$3(V_2Si + Fe_2Si) + 8VOF_3 = 4VO_2 + 2Fe_3V_5 + 6SiF_4$$
$$V_2Si + Fe_2Si + 4PbF_2 = 2FeV + 2SiF_4 + 4Pb.$$

In the latter case lead itself volatilizes from the furnace. These reactions of the fluorides have not yet been practically utilized.

PREPARATION OF NICKEL VANADIUM.

This is prepared in a similar manner to ferro-vanadium. A cast carburized alloy of nickel and vanadium is made by reducing V_2O_5, mixed with nickel oxide, by carbon, according to the reaction:

$$2V_2O_5 + NiO + 15C = 4VC + Ni + 11CO.$$

The carburized metal is refined by the further use of nickel oxide or by vanadic acid.

$$4VC + Ni + 4NiO = Ni_5V_4 + 4CO.$$

Another method is to produce first double silicide of nickel and vanadium and remove the silicon therefrom by a mixture of vanadic acid and lime, according to the reaction:

$$V_2O_5 + 2NiO + 2SiO_2 + 11C = V_2Si + Ni_2Si + 11CO.$$
$$3(V_2Si + Ni_2Si) + 4V_2O_5 + 6CaO = 6NiV + 6CaSiO_3 + 4V_2O_2.$$

The above reactions are performed in the same furnace and under the same conditions as the manufacture of ferro-vanadium.

PREPARATION OF FERRO-NICKEL-VANADIUM.

This may be obtained by the direct fusion of ferro-vanadium with metallic nickel, or by the refining of carburized ferro-vanadium by nickel oxide, or by the refining of the double silicide of iron and vanadium by nickel oxide, or by the refining of the vanadium silicide VSi_2 by a mixture of the oxides of iron and nickel, in the presence of lime.

$$VSi_2 + Fe_2O_3 + NiO + 2CaO = 2CaSiO_3 + Fe_2NiV.$$

The metallo-thermic reaction using aluminium may also be used, but is not so practicable.

PREPARATION OF FERRO-CHROME-VANADIUM.

This may be obtained by the metallo-thermic reaction using aluminium, but at a rather high cost. A better method is to produce double silicide of chromium and vanadium, which is then

refined by a mixture of iron oxide and lime. The double silicide is produced by the following reaction:

$$V_2O_5 + 3Cr_2O_3 + 5SiO_2 + 24C = 2VSi + 3Cr_2Si + 24CO.$$

The refining of the double silicide is carried out in accordance with the following reaction:

$$FeSi + (2VSi + 3Cr_2Si) + 4Fe_2O_3 + 6CaO = 6CaSiO_3 + Fe_9Cr_6V_2.$$

In the sample given an alloy was obtained containing

Vanadium	11.3%
Chromium	33.9
Iron	54.8

This composition is quite suitable for industrial purposes.

PREPARATION OF FERRO-TUNGSTEN-VANADIUM.

This may be obtained easily by the metallo-thermic reactions using aluminium, but at a high cost.

Gin has devised the following metallo-thermic reaction using silicon. The double silicide of tungsten and vanadium is first produced by reduction by carbon, which is then refined by a mixture of oxide of iron and lime according to the following reaction:

$$V_2O_5 + 6WO_3 + 11SiO_2 + 45C = 2VSi + 3W_2Si_3 + 45CO.$$
$$Fe_2Si + (2VSi + 3W_2Si_3) + 8Fe_2O_3 + 12CaO = 12CaSiO_3 + 2Fe_9W_3V.$$

The above operation has given the author an alloy containing:

Vanadium	4.7%
Tungsten	50.2
Iron	45.1

PREPARATION OF CUPRO-VANADIUM.

The direct manufacture by metallo-thermic reaction, using aluminium, does not give good results. It is obtained much more easily by refining the silicide of copper oxide. After manufac-

turing the silicide V_2Si, it is melted with an excess of copper and then small portions of a mixture of oxide of copper and lime are thrown on the surface of the bath. The reaction is almost instantaneous, forming a calcium silicate:

$$(10Cu + V_2Si) + 2CuO + CaO = CaSiO_3 + 2Cu_6V.$$

PREPARATION OF FERRO-VANADIUM BY ALUMINO-THERMY.

The great heat of oxidation of aluminium is utilized by Dr. Goldschmidt, of Essen, using pulverized aluminium. He has thus isolated chromium and manganese almost pure. When using melted aluminium it is only possible generally to obtain alloys. The reaction:

$$3V_2O_5 + 10Al = V_6 + 5Al_2O_3,$$

has not been realized. By using iron oxide at the same time with the vanadic acid the reaction takes place, yielding a certain proportion of reduced vanadium, the reaction being:

$$3V_2O_5 + 9Fe_2O_3 + 28Al = 6Fe_3V + 14Al_2O_3.$$

It is scarcely possible to obtain more than 27 percent vanadium in this alloy. Captain Nicolardot gives the following description of the method.[2]

A large crucible of refractory earth or plumbago is lined inside with magnesia. Captain Caron[3] has shown how to get a solid brasque of this material.

The lining is thickest at the bottom of the crucible.

The crucible is put inside a layer of carbon, surrounded by refractory bricks. The space between is filled with small pieces of coke, forming an insulating envelope, protecting the crucible from loss of heat by radiation and conduction and currents of air which may crack it. A funnel is placed to one side, in which is the material to be reduced.

The ignition of the mixture is a delicate operation, necessitating a high initial temperature, which is obtained by a fuse of sodium peroxide and aluminium or magnesium. This is prepared just at the time it is required for use, because the sodium peroxide

[2] Revue d'Artillerie, March, 1904.
[3] Compt. rend, 66, 839 (1868).

is very deliquescent. This mixture burns more easily than the barium peroxide fuse. The mixture is ignited by a red-hot iron rod.

The reaction takes place with great violence. As soon as it

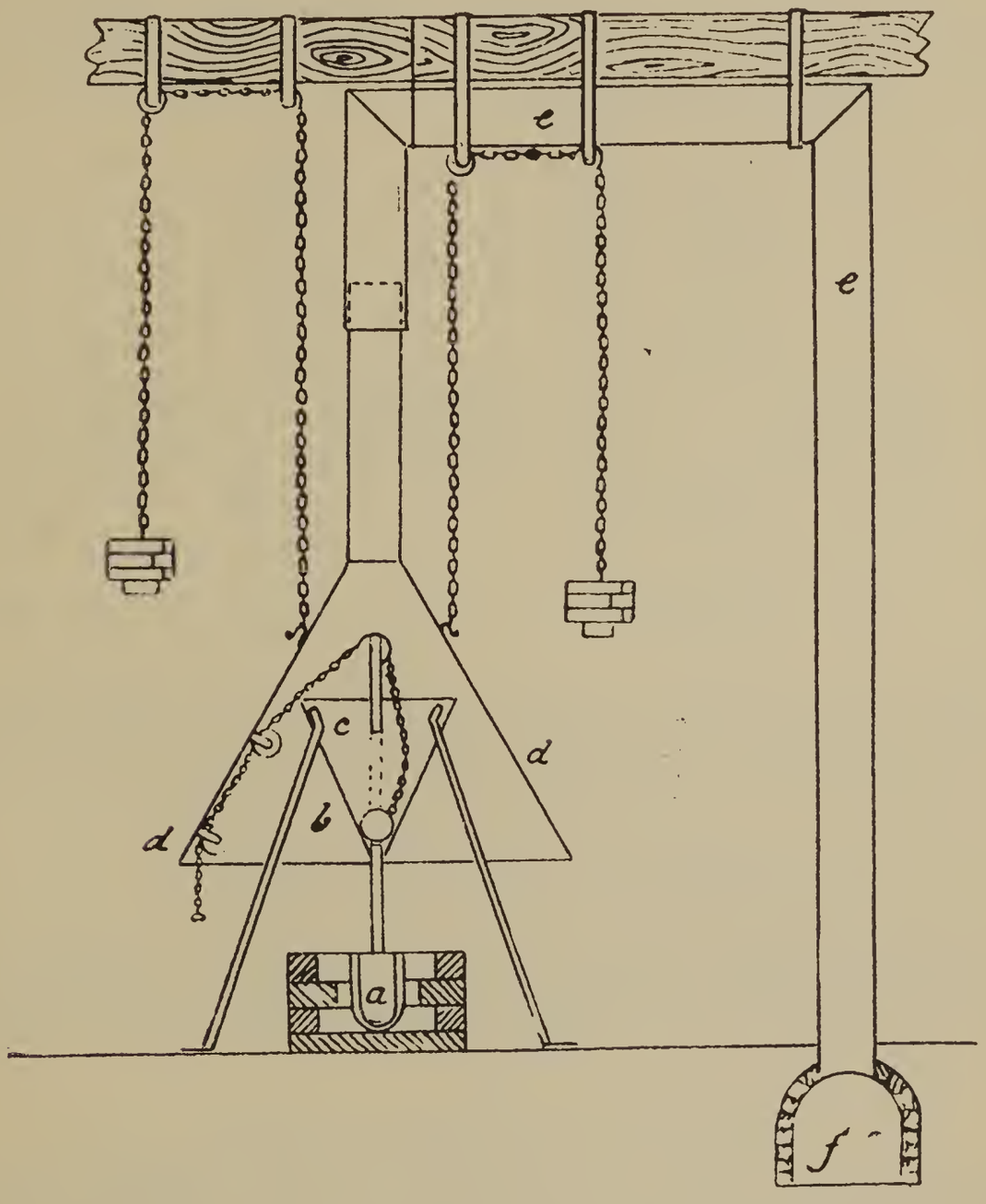


FIG. 4.

subsides more mixture is run in and the operation continued. At the end of about five minutes the crucible is filled with the mixture of slag and alloy, and the operation is terminated. The alloy is tapped out from the bottom of the crucible, and if the lining is still solid a further quantity may be reduced in the same crucible, which is rarely the case.

The small ingot obtained is brilliant white, with slightly yellowish reflections like antimony; it is quite different from the metal obtained in the electric furnace. The slag is vitreous and vesicular. It is extremely hard, easily scratching glass.

In some tests made by Mr. Proctor Smith and published by him, a mixture of 15 parts oxide and 4.5 parts aluminium in grains was put into the crucible. The mixture of oxides used had the following composition:

Vanadic acid	16.6%
Ferric oxide	58.2
Silica	1
Sodium salts	18.2
Moisture	5.6

The alloy obtained had the composition:

Vanadium	14.9%
Iron	58.1
Aluminium	26
Silicon	1.3

The alloys obtained in practice may contain very little aluminium if the charged is proportioned according to the following reaction:

$$3V_2O_5 + 10Al = 6V + 5Al_2O_3$$
$$Fe_2O_3 + 2Al = 2Fe + Al_2O_3.$$

When the mixture being reduced is too rich in vanadium it may be diluted by the addition of mill scale, the reduction of this oxide taking place as follows:

$$3Fe_3O_4 + 8Al = 9Fe + 4Al_2O_3.$$

For example, to obtain an alloy of 25 percent vanadium, using 75 percent vanadic oxide, one may use 6 parts of the mixture of oxides, 9 parts of mill scale and 25 parts of aluminium. To avoid having too much aluminium in the alloy, the reduction may be less complete and the slag contain more vanadic acid, which can be made more fusible by the addition of sodium carbonate. In fact, alloys as low as 2 percent aluminium may be obtained.

At the present time the V_2O_5 to be used for the reduction by

aluminium is used in the form of vanadate of iron containing 75 percent V_2O_5, and corresponding to the formula FeV_2O_6. When this compound is treated the reaction would be as follows:

$$6FeV_2O_6 + 15Fe_2O_3 + 27Al_2 = 12Fe_3V + 27Al_2O_3.$$

A little sodium oxide may be added to the mixture to make the slag more fluid.

COST OF FERRO-VANADIUM REDUCED BY ALUMINIUM.

In 1907 the cost of thus producing 100 kilograms of 25 percent ferro-vanadium was as follows:

Raw Materials:		
60 kg purified V_2O_5 @ $1.92	$115.20	
52 kg pulverized aluminum @ $0.50	26.00	
90 kg mill scale @ $0.04	3.60	
Magnesia bricks and powder	5.00	
Miscellaneous	3.00	
		$ 160.00
Fuel for drying products		1.00
Labor		8.00
General and miscellaneous expenses		11.00
		$180.00

That is, $1.80 per kilogram of 25 percent vanadium alloy, or $7.20 per kilogram of contained vanadium.

Vanadic acid can be obtained to-day cheaper than this, and it is probable that the alumino-thermic method should be able to manufacture 25 percent alloy at $1.50 per kilogram; this price is still high, and will prevent ferro-vanadium attaining the large usage which it otherwise might reach.

MODIFICATION OF THE METALLO-THERMIC PROCESS.

The reducing action of aluminium and carbon may be simultaneously utilized in the electric furnace, by introducing a mixture of vanadic acid and carbon, and when the reduction has reached a certain point, projecting into the bath powdered aluminium to finish the reduction. Ferro-vanadium can be easily obtained in this way, using the proportions given by the formula:

$$2(FeO.V_2O_5) + 10Fe + 9C + Al_2 = 4Fe_3V + Al_2O_3 + 9CO.$$

Nevertheless, the manufacture of pure vanadium in this way

is impossible; there results at best cast vanadium containing 5-6 percent carbon.

Twenty-five percent ferro-vanadium containing 1-1.5 percent carbon may be prepared by performing the above reduction in a bath of melted soft iron. Some lime and calcium fluoride may be used to make a more fusible slag.

This mixed method is very much more economical than the Goldschmidt process, for the consumption of aluminium is lessened two-thirds or more.

MIXED PROCESS OF HELOUIS.

Helouis has patented[4] a modification of the Goldschmidt process, consisting in performing the reduction by a mixture of sodium and aluminium powder, The slag is a thin and very fusible mixture of aluminate of soda. The same end may be attained by adding carbonate or oxide of soda to the aluminium powder.

EXPERIMENTS OF GIN.

Gin has tried the reduction of vanadium oxides by a mixture of aluminium powder and magnesium wire. The results are better than with vanadium alone, but the cost is increased. Ferro-vanadium, 31 percent V., has been thus obtained.

Gin has also tried the reduction with aluminium powder and electrolytic calcium, but by this method was not able to obtain ferro-vanadium with more than 30 percent vanadium.

Gin has likewise attempted the reduction by a mixture of aluminium powder and 60 percent ferro-silicon, pulverized. The reaction takes place approximately according to the following proportions:

$$27V_2O_5 + 43Fe_2O_3 + 22FeSi_3 + 66Al_2 = 54Fe_2V + 66Al_2SiO_5.$$

Practically the following proportions were used:

Vanadic acid	50 kg
Oxide of iron.....	70 "
60% Ferro-silicon	30 "
Aluminium	40 "

The ferro-vanadium obtained contained 25-30 percent V.

[4] French patent No. 252,973, 17 April, 1906.

ELECTROLYTIC PREPARATION OF VANADIUM.

ELECTROLYSIS OF AQUEOUS SOLUTIONS.

Cowper-Coles obtained a brilliant metallic deposit by electrolysis of a solution of vanadic acid in the presence of sodium chloride. He worked at 80° C., with a current of two amperes per square centimeter and an electromotive force of two volts. On repeating this experiment, I have observed that the metallic deposit soon stops, tarnishes, and does not exceed a very limited thickness. It seems that the deposit is protected at first by a film of hydrogen, which soon loses its efficacy.

In the present state of the experiments it is hardly possible to believe that vanadium can be manufactured industrially by the electrolysis of aqueous solutions.

ELECTROLYSIS OF IGNEOUS ELECTROLYTES.

Gin Process.

Gin electrolyzes V_2O_3 or V_2O_5 dissolved in a bath of molten tetra-fluoride of vanadium and fluoride of calcium made up according to the following formula: $2V_2F_4.CaF_2$. The anode is carbon and the cathode iron, nickel, copper or lead, according as one wishes to obtain ferro-vanadium, nickel-vanadium, cupro-vanadium, or pure vanadium. In the latter case the lead is subsequently eliminated from the alloy by volatilization in the electric furnace. When the cathode is of copper or lead, a quantity of potassium fluoride may be added to the bath to reduce the melting point of the electrolyte so as not to volatilize the cathode metals. The electrolyte is replenished with pulverized V_2O_4. Metallic vanadium cannot be directly obtained; its melting point is above the boiling point of the electrolyte. This process presents analogies with that of Minet and the Heroult-Hall processes for aluminium.

With a current density of 1.2 amperes per square centimeter of anode, and 2 amperes per square centimeter of cathode, the bath can be kept fused by the resistance heat and the electrolysis performed with an electro-motive force of 10-12 volts. The melting point is above 1100°; its resistance when melted is about 0.4 ohms/cm./sq. cm., which is reduced by the addition of V_2O_4.

The electrolyte is made of a mixture of pure lime and tetra-

oxide of vanadium in the proportions: $2V_2O_4 + CaO$, and treated with concentrated or gaseous hydrofluoric acid; the excess of acid used is saturated with a supplementary addition of lime, the solution is concentrated, dried, and calcined at low redness.

Second Gin Process.

This method rests on the high conductibility of V_2O_3 and on the facility with which vanadium tri-fluoride can be obtained by attacking the tri-oxide by fluorine in the presence of carbon. The vanadium tri-oxide is obtained from vanadic acid by calcination of a mixture of it and carbon; it is then mixed with the proper proportion of carbon for the reaction:

$$V_2O_3 + 3C = 2V + 3CO.$$

This mixture is moulded into an electrode which is to be used as anode. The specific resistance of such an electrode, as measured at the Central Electrical Laboratory in Paris by Chaumat and Petitalot, was 0.0137 ohms/cm./ sq. cm., at 15°.

Such electrodes will support a current density of three-fourths that which is used for a carbon electrode of the same section. A bundle of such electrodes is used as anode against a cathode formed of a block of steel.

The electrolyte used is at starting a solution of ferrous fluoride in calcium fluoride. This melts at a low temperature, and on the passage of the current the fluoride liberated at the anode reacts upon it to form vanadium tri-fluoride, while the carbon abstracts the oxygen according to the formula:

$$V_2O_3 + 3C + 6F = 2VF_3 + 3CO.$$

The vanadium fluoride passes into the electrolyte and is subsequently decomposed by the current, which deposits at the cathode both iron and vanadium. To obtain ferro-vanadium with more than 25 percent vanadium, the cathode surface should be less than the active surface of the anode. A sufficiently fluid bath is obtained with a current density of 2 amperes per square centimeter of anode and 6 amperes per square centimeter of cathode, the voltage running between 11 and 12 volts.

The ferrous fluoride is only intended to start the operation, but since a small quantity of fluorine is lost by the formation of CF_4

at the anode, this loss of fluorine may be made by the addition of some more ferrous fluoride to the bath from time to time.

After each tapping of ferro-vanadium fresh iron is introduced into the bath and melted; it is nearly useless to try to obtain pure vanadium, because it taps with great difficulty, and can practically only be extracted from the furnace in a solid state.

With modifications this process may be used for manufacturing other alloys of vanadium, such as with copper, manganese, etc.

USES OF VANADIUM.

USES IN INDUSTRIAL CHEMISTRY.

Berzelius was the first to use vanadium industrially, in the manufacture of a black ink containing tannic acid. Böttger obtained a better ink by making vanadium pyrogallate. In 1861 St. Claire Deville proposed to use vanadium in ceramic decoration. At that time no success attended this attempt, but it has been since taken up at the Royal Porcelain Factory at Copenhagen, and more recently ceramic effects worthy of attention have been thus obtained in France.

Vanadium tetroxide or the purple intermediate oxide have likewise been employed as metallic paints under the name of vanadium bronzes.

In glass, vanadium gives very fine browns, purples, blues and greens. This application has been studied by Messrs. Cooper-Cowles & Co., of London.

The most important use of vanadium is in the dyeing industry. The ease with which vanadium compounds absorb and give up oxygen permits of using the compounds as oxidizing or reducing agents, or even as such alternately.

It has been particularly noted that the compounds of other acids with vanadic acid react in acid solutions on the salts of anilin to give rise to a very dark dye only slightly soluble in most solvents. In this condition the vanadium acting in the presence of an oxidizing agent abstracts hydrogen from the anilin.

I. Lightfoot was the first to show in 1871 the remarkable properties of vanadium as an oxidizer of printing inks. He announced

that it acted much more quickly than other oxidizers and gave richer blacks.

Antoni Guyard showed subsequently the extraordinary influence of the chlorhydrate of the tetroxide of vanadium, one part of which suffices to transform 400 parts of anilin into anilin black.

The vanadium plays the part of an intermediate transporting agent for the oxygen from the oxidizing agent to the anilin.

Anilin black is generally formed directly on the fibre. The oxidizing agent is sodium chlorate, and the vehicle is the chloride of vanadyl, or vanadic acid. The vanadium compounds also have the valuable property of not attacking the rolls of the printing press. They make very rich blacks, more solid, and give finer impressions. According to the duration of the oxidizing influence the following colorations may be obtained: greenish, emerald, nigraline or anilin black properly so called, and dead black.

The dyeing of vegetable fibres with chlorate baths containing vanadium presents no difficulties. Baths for silk should contain 30 to 50 grams of gum arabic per litre. Wool should be previously freed from grease, and the bath should contain 2 to 3 times as much anilin as for cotton. The addition of a little hydrochloric acid has proven advantageous up to 10 grams per litre.

The vanadium dye with anilin black gives very fine blacks without altering the tissues and with great rapidity of action and ease of manipulation. The tissue is saturated in a bath of anilin chlorhydrate, chlorate of potash and the vanadium salt. The oxidation is produced by steam drying; the development and fixation of the black is effected in a chromium bath.

Pinckney recommends the following proportions:

Anilin chlor-hydrate....	550	parts
Vanadium salts	18.5	"
Nickelous chloride	20	"
Potassium chlorate.....	100	"
Water 2,500		"

For the dyeing of wool, Hommey recommends:

Water	1,000	grams
Analine chlor-hydrate	80	"
Potassium chlorate	40	"
Hydrochloric acid	5	"
Ammonium vanadate	0.91	"

Prudhomme recommends the following proportions for the baths made up in the following order:

Sodium chlorate	2,300	grams
Water	20	litres
Analine salt	4,000	grams
Anilin oil	150	grams
Water	8	litres

Next:

Yellow Prussiate	2,500	grams
Water	14	litres

Finally:

Chloride of vanadium, 1%	300	ccm.

Make the total up to 50 litres with water.

The pieces are passed through this bath, dried at a hot flue and steamed for three minutes. The yellow color may appear during the steaming; one terminates the steaming by passing through a 5 percent bath of chromate at 60°, followed by washing.

The vanadium salts may likewise be employed in printing, and Pinckney proposes the following proportions:

Anilin chlor-hydrate	150	parts
Vanadium salts	18.5	"
Nickelous chloride	20	"
Sodium or Potassium chlorate	100	"
Water	1,200	"

This is thickened up with gum or dextrine.

Witz uses for printing 0.0013 of vanadium to 1 litre of color, while the ordinary bath requires 500 grams of sulphide of copper to 1 kilogram of anilin.

There is used more nearly in the formula of the Höchst Color Works:

Anilin salt	12	grams
Analine	18	"
Sodium chlorate	35	"
Starch thickening	875	"
Vanadium solution, 2%.....	2	"

replacing 90 grams of pasty sulphide of copper.

In the French Patent No. 270,552, September 17, 1897, the Société des Mines de Yalui patented a dyeing process for vegetable fibres, using the vanadium salts as follows:

1. Vanadium salts for transforming the surfaces of the fibre into oxy-cellulose.

2. Vanadium salts as re-agents in the dyeing bath.

Photographic Propertics of Vanadium Salts.

The Lumiere Brothers have studied this reaction. By reducing the salts of V_2O_5 in an acid, as for instance H_2SO_4, by zinc, the vanadium liquor becomes at first red, then blue, green and finally violet, corresponding to the successive formation of red vanadic sulphate, blue hypo-vanadic sulphate and violet hypo-vanadous sulphate. In the last condition the solution is an energetic developer, acting even when strongly acid.

Aside from utilizing these hypo-vanadous compounds for developing the latent photographic image the vanadic salts are of interest as being capable of giving a photographic image by reduction under the influence of light. V_2O_5 dissolves in a number of acids, furnishing red or yellow unstable solutions, which are generally decomposed by water. Amongst these the chloride, phosphate and potasso-tartrate have given the best results regarding their sensibility to light.

They have been prepared from V_2O_5 by treating it with cold concentrated hydrochloric acid. The red solution is dissociated by water in small quantity; it is therefore not diluted before being used. If necssary to dilute this may be done by a mixture of alcohol and water, which avoids the dissociation. If a sheet of gelatine paper is impregnated with this liquid it is necessary to dry in the dark, for the substance is reduced by light. On exposing the paper, thus sensitized, under a positive, one obtains a

proof which can be accentuated and fixed by the aid of aromatic amines.

Vanadic phosphate is obtained by heating V_2O_5 with P_2O_5 to 150°. It is less likely to dissociate on contact with water, but it is less sensitive to light than the chloride.

The potasso-tartrate is obtained by shaking V_2O_5 with a solution of bi-tartrate of potassium. The yellow solution thus obtained, cold, gives papers which are very sensitive, and may be preserved without alteration for several weeks.

The color reactions obtained with these several preparations are few and the images very feeble. These defects may be attributed properly to the relatively feeble oxidizing power of the vanadium salts.

Use of Vanadium in the Contact Process.

De Haen has patented the use of V_2O_5 as a catalytic agent for the manufacture of SO_3 by contact.

Helouis has patented, under the name of the Société Anonyme des Mines de Yauli, French Patent No. 292,992, October 2, 1899, a process for the manufacture of anhydrous acids by the use of V_2O_5 or its compounds. He treats the sulphuric acid coming from the lead chambers, at 55° B., with red V_2O_5 so as to obtain the sulphate, $V_2O_5.3SO_3$. This trisulphate can be completely dehydrated at a temperature of 300°. By raising the temperature to 650° it is dissociated into V_2O_5 and anhydrous SO_3, the process thus being cyclic. In place of V_2O_5, sodium meta- vanadate containing 30 percent Na_2O and 70 percent V_2O_5 may be used, absorbing in this case five equivalents of SO_3 and producing a mixture of sodium bi-sulphate and vanadium tri-sulphate $Na_2O.2SO_3 + V_2O_5.3SO_3$. This mixture may be dehydrated at a low temperature and then decomposed at a high temperature, giving off all its SO_3 and leaving behind $Na_2O.V_2O_5$ to be used over.

This process permits of obtaining anhydrous or fuming sulphuric acid, or even pure SO_3. Other compounds of vanadium may be used in the same manner. The process may likewise be used for the manufacture of anhydrous phosphoric acid and other similar acids.

Gin has noticed, on repeating these experiments, that they

give good results only by passing the products of the decomposition over platinized asbestos, mixing them with some air, at a temperature of 400°, so as to counteract the tendency of SO_3 to dissociate into SO_2 and O at the temperature at which it is evolved from the decomposed compounds.

Applications to Biological Chemistry.

Ferro-vanadium has been used in the preparation of filters for purifying water. The vanadium oxidizes organic compounds and it reoxidizes automatically on exposure to the air, without agglomerating or stopping the filter.

Calcium vanadate is assimilated by plants and by animals, and in the organism plays a part similar to calcium phosphate. Vanadiol has been employed in therapeutics; it is composed of peroxide of vanadium and has been used for destroying the Koch bacillus.

Use in Electric Lighting.

Vanadium has been proposed as a material for incandescent lamp filaments, and the French Thompson-Houston Company has patented a process of making vanadium carbide, pulverizing and agglomerating it for the manufacture of filaments. The filaments embedded in powdered graphite are heated in a graphite crucible in a vacuum furnace to a temperature of 2000-2400°.

USE OF VANANDIUM IN STEEL.

Vanandium enters into steel not only as an ameliorator of carbon steels, but it also purifies the metal from dissolved gases, notably from nitrogen and oxygen, furnishing thus a metal of remarkable homogenity.

The presence of vanadium in the ores used for making the best Swedish iron suggested the important role which vanadium may play in the metallurgy of steel. The first definite experiments were made at Firminy in 1896 by Choubley. Vanadium was added to steel in the form of a mixture of aluminium powder and vanadium oxides. After many unfruitful attempts, a steel was obtained with a considerable increase in resistance to shock. Seven blows were necessary to break the steel as against three blows for the ordinary steels. The test of "the mandrel" showed

still more remarkable steels. The mandrel slid 154 mm. with the rings of vanadium steel, and even traversed one completely without breaking it, while the sliding with ordinary steels was only 51 mm.

Finally, the tensile tests gave the following results:

	Ordinary Steel	Vanadium Steel
Elastic limit (per sq. mm.)	75.6 kg	109.5 kg
Tensile strength (per sq. mm.)	87.6 "	115.2 "
Elongation	10 %	11.3%
Contraction of area	54 "	47 "

The increase of elastic limit was thus 44.8 percent, and of tensile strength 31.5 percent, while the elongation and contraction did not appear to diminish.

These very encouraging experiments showed that vanadium improves ordinary steel, in hardness and ease of tempering. The elastic limit and tensile strength increase in great proportion, while the elongation and contraction remain sufficient for practical purposes. It is particularly advisable to incorporate vanadium in low carbon steels containing a minimum of aluminium and silicon. Choubly concluded to use high ferro-vanadium alloy for adding vanadium in small proportions to the steel.

Vanadium steels have the remarkable property of hardening very appreciably with increase in temperature. This property opens a considerable field for their employment in the manufacture of bearings, shells and high speed tools.

Experiments of Helouis.

The same year, 1896, Helouis described to the "Société d'Encouragement" the important role of vanadium in metallurgy. The steel used by Helouis was made by Marchal, superintendent of the forge at Pantin. Tests made in a graphite crucible showed no improvement of the steel by the introduction of vanadium. Tests were then made in magnesia crucibles, since it was thought the first failure was due to the carburization of the steel. In these crucibles was obtained a steel having 60 percent increased tensile strength.

Analogous experiments were made at the Ecole des Mines.

Unfortunately the exact chemical analyses of these steels were never published.

Experiments of Arnold.

In 1900 the more exact researches of Professor Arnold at Sheffield were made, using pure vanadium made by the Goldschmidt process and furnished by the New Vanadium Alloys Company. Six ingots were made in crucibles by the ordinary process, one from Swedish iron and the other two from Bessemer metal made from Swedish iron. These ingots averaged: Si 0.1 percent , S 0.35 percent, P 0.015 percent, Mn 0.05 percent. The vanadium used was in the form of ferro-vanadium, containing a little aluminium. The ingots were rolled to ¾-inch round bars, which were then tested. Some aluminium and vanadium were lost during the melting, and when they were added to the bath before melting the larger part of the aluminium and approximately 30 percent of the vanadium were lost by oxidation. When the alloy was added 15 minutes before casting, all the vanadium and aluminium added passed into the steel. One deduction from this fact is that the percentage of aluminium in the alloy used must necessarily be low, that is, below 1 percent.

With Swedish pure iron the addition of 0.85 percent vanadium increased the elastic limit 12.88 kilograms per square millimeter (18,400 pounds per square inch) and tensile strength 8 kilograms (11,400 pounds), without sensibly diminishing the ductility. The influence of vanadium on soft iron is far inferior to its extraordinary influence on steel.

Almost pure steel, with 1.1 percent carbon, was treated with 0.14 percent vanadium, and without sensibly changing the ductility its elastice limit was increased 20.48 kilograms (29,200 pounds) and its tensile strength 12.2 kilograms (17,400). Likewise the addition of 0.3 percent vanadium to the same steel without diminishing its ductility increased the tensile strength to 119.6 kilograms (170,800 pounds), and 0.6 percent vanadium increased it to 134.3 kilograms (192,000 pounds).

There seems to be no change on adding more than 0.6 percent vanadium. With this amount the elastic limit is nearly 102 kilograms (145,000 pounds).

Investigations of L. Guillet.

Professor Guillet, of the "Conservatoire des Arts et Metiers," undertook a systematic study of the micrography of the vanadium steels. A complete statement of his conclusions was presented to the Congress of Metallurgy at Liege, in June, 1905, and has appeared later in the "Revue de Metallurgie."

His results have shown very conclusively that vanadium steels containing over 0.7 percent vanadium contain vanadium carbides, and have no important industrial properties. The only vanadium steels of practical use are, therefore, those containing less than 0.7 percent vanadium. The influence of vanadium is to harden steel, to increase its elastic limit and tensile strength; it plays a role analogous to carbon, but with less intensity. But vanadium possesses an entirely different characteristic, namely, that the increase of mechanical strength is not accompanied with a decrease of elongation or ductility, and fragility is not induced.

The employment of vanadium steels will largely depend on the cost of vanadium. A short time ago it cost $10.00 per pound for vanadium in ferro-vanadium. At present an American company sells it for $5.50 a pound. When it is remembered that the most interesting of the vanadium steels contain only 0.2 to 0.5 percent vanadium, the increase of price above that of carbon steels is only 1 to 2½ cents per pound; the loss during addition may be neglected.

Our present opinion is that vanadium steels with low carbon content contain a double carbide of the form $mFe_3C + nV_2C$, the eutectic of this containing m greater than n. Above a certain limit all the vanadium is in the state of carbide which is insoluble in iron, either α, β or γ iron. It is possible that this carbide does not even dissolve in molten iron at a high temperature. Later researches of Mr. Guillet in 1904 and 1907 confirm this point of view. Guillet's detailed communication to the Academy of Sciences may be found in *Comptes rendus*, August 8, 1904, and *Revue de Metallurgie*, August, 1907.

Researches of Putz.

An interesting study was published in 1907 in the German journal, "Metallurgie," by Putz, describing his work on the

complex vanadium steels. Particular attention is called to the nickel-vanadium and tungsten-vanadium steels. The important industrial conclusion of Mr. Putz is to the effect that the nickel-vanadium steels should be restricted in composition to the following limits:

Carbon	0.1-0.3%
Nickel	2 -7
Vanadium	0.1-0.3

Use of Vanadium Silicide in Cast Iron.

Gin introduced this pulverized silicide into the runner. Its addition through the cupola is impossible because of the oxidation of the vanadium. The silicide used contained silicon 21.14 percent, vanadium 24.16, iron 53.88, carbon 0.52. A uniform 3 percent of this silicide was added to different kinds of iron, in each causing sensible increase of tensile strength and resistance to shock. Highly carburized cast iron becomes very brittle; less carburized becomes softer and much stronger.

Moldenke has noted that the addition of 0.05 percent of vanadium was sufficient to restore burned iron and double its strength.

Method of Using Vanadium in Steel.

In his interesting work on vanadium, Nicolardot has formulated as follows his experiences on incorporating vanadium into steels:

The great affinity of vanadium for oxygen and carbon must be kept in mind. In general, precautions such as are used for introducing chromium into steel should be observed, but even more rigorously in the case of vanadium, since it is more oxidizable than chromium. The atmosphere of the furnace or of the crucible should be neutral or reducing, and the steel should contain as little oxygen as possible when the vanadium is added. Oxygen transforms vanadium very rapidly into vanadic acid, which melts at a low temperature and produces very fluid slags which clean the steel. This is an interesting phenomenon, but it is attaining, by means of a very expensive reagent, the same effects as aluminium, which added in very small quantity produces it very much cheaper.

Vanadium steels cannot be produced in the Bessemer converter

or the open-hearth furnace; the crucible process is the only one admissible. [The electric furnace is equally available.—*Translators.*]

The first test at Firminy showed that vanadium should be used as an alloy rich in vanadium. This alloy was melted by itself in a magnesia crucible and poured into the steel at the last moment.

If ferro-vanadium is added with the charge, at least one-third of the vanadium is lost by oxidation, and almost all the aluminium. On the contrary, if ferro-vanadium is melted and poured into the steel ten to fifteen minutes before removing from the furnace, all the vanadium and aluminium are incorporated in the steel.

It is also possible to prepare vanadium steels by the method of Professor Arnold, who took melted steels coming from the Martin furnace, caught them in a ladle previously highly heated by a coke fire, and a few minutes before casting poured into the steel in the ladle melted ferro-vanadium from a crucible. One can thus treat several hundred kilograms of steel in the ladle. The chief difficulty of this method is the fusion of the alloy, there being considerable loss in the crucible in which it is melted, and also in the transfer to the steel. This loss, being irregular, causes great variations in the proportions of vanadium which enters the steel. There is still great room for improvement in the methods of introducing vanadium into steel.

Ferro-vanadium has been manufactured with 30 and even 50 percent vanadium, aluminium below 2 percent, and silicon below 1 percent. Ferro-vanadiums prepared in the electric furnace are the richest in carbon, up to 8 percent, while that prepared by the Goldschmidt method with aluminium contains practically no carbon.

To the above observations of Mr. Nicolardot we may add that the use of ferro-vanadium is particularly easy in the electric furnace, where one is operating in a neutral atmosphere and a perfectly closed space. Under these conditions there is no loss of vanadium by oxidation from the air, and only oxidation by the oxygen in the molten bath. The vanadium deoxidizes the bath, producing a very liquid slag, which rises to the surface and is eliminated with extreme quickness, leaving a metal perfectly pure; in this respect it differs radically from aluminium.

The formation in the fused steel of carbides of vanadium which

are insoluble in the iron and less fusible than it, constitutes the greatest obstacle to the use of vanadium in the manufacture of crucible steels. In fact, in the crucible the steel dissolves from the vessel some of the graphite, so that it is thus impossible to properly melt the steels. Under these conditions the formation of these vanadium carbides is unavoidable, and there result steels of heterogeneous structure.

The tests so far made show that vanadium steels should contain a minimum content of carbon, the necessary hardness being obtained by silicon, chromium, nickel, tungsten and molybdenum.

It would be very interesting if metallurgists would prepare ferro-vanadium or vanadium silicide or combinations of vanadium with nickel, chromium, tungsten and molybdenum free from carbon for use in these steels.

ANALYTICAL CHEMISTRY OF VANADIUM.

HYPO-VANADIC SALTS (*vanadium as a base*).

These salts are brown in the anhydrous condition, and when hydrated or dissolved, green or blue. The hydrochloric acid solutions give the following reactions:

Alkalies: A small amount of alkali gives a green precipitate, soon becoming brown. A slight excess of the reagent dissolves the precipitate, forming a brown solution containing hypo-vanadate. A large excess of alkali gives a brown precipitate, the solution remaining brown because the hypo-vanadate is slightly soluble in excess of alkali. If the precipitate is washed with pure water, it dissolves to a brown solution.

Alkaline Carbonates: The reactions are the same as for alkali.

Alkaline Bicarbonates: They produce at first a brown precipitate, soluble in excess of the reagent. The liquid becomes cloudy on boiling, and deposits a large part of its hypo-vanadates, leaving, however, a brown solution.

Ammonia: This gives at first a brown solution. A larger excess of ammonia gives a brown precipitate, supposed to be ammonium hypo-vanadate. This precipitate is somewhat

soluble in water, but precipitation is complete in the presence of ammonia and ammoniacal salts.

Hydrogen Sulphide: This gives no precipitate in a strong hydrochloric acid solution of V_2O_4 or of $VOCl_2$; when the solution is dilute and contains metals precipitated by hydrogen sulphide, such as lead or copper, there is a partial precipitation of vanadium.

Ammonium Sulphide: Precipitates at first a brown sulphide which is easily soluble in excess of the reagent; the dark red precipitate is a sulph-hypo-vanadate. Traces of vanadium give a purple coloration. These solutions are very stable and do not give a precipitate of vanadium sulphide until after complete decomposition of the alkaline sulphide.

Potassium Ferro-cyanide: A greenish-yellow precipitate, slowly becoming green in the air.

Potassium Ferri-cyanide: A yellowish-green precipitate.

Tincture of Galls: Gives a blue-black precipitate comparable to a kind of ink.

Organic materials hinder precipitation by the alkaline carbonates, alkalies and ammonia, but do not interfere with the reactions of hydrogen sulphide or ammonium sulphide.

VANADIC SALTS (*vanadium as base*).

The solutions are yellow or red.

The hydrochloric acid solutions when heated slightly disengage chlorine and become green. A more rapid reduction is made by reducing agents, such as sulphurous acid, hydrogen sulphide, alcohol, sugar, oxalic and tartaric acids, or by sheet zinc. There is then obtained a blue solution due to the formation of hypo-vanadic salt.

HYPOVANADATES (*vanadium as an acid*).

These salts are brown, soluble in water, slightly soluble or insoluble in alkalies and alkaline salts.

The insolubility of ammonium hypo-vanadate in ammonia and concentrated solutions of ammonium salts is utilized for the estimation of vanadium.

Air: Oxidizes solutions of these salts and decolorizes them.

Hydrogen Sulphide: Forms red sulpho-salts.

Alkali Sulphides: Produce soluble red sulpho-salts.

VANADATES (*vanadium as acid*).

Alkaline vanadates are soluble in water. Ammonium meta-vanadate is insoluble in strong solutions of ammonium chloride; this property is used for separating out vanadic acid. Vanadates are only slightly soluble in alcohol and alkaline solutions.

Metallic vanadates are very slightly soluble in water, but easily soluble in acids, especially in nitric acid, which liberates vanadic acid without decomposing it.

Analytical characteristics: Before the blowpipe, vanadium salts give characteristic tests when they are free from metallic oxides, in particular iron. Heated alone in the reducing flame on charcoal, they are reduced to black trioxides; in the oxidizing flame they pass to the condition of dark brown pentoxides. Fused with soda, they form a bead, brown in the reducing flame, and greenish in the oxidizing flame; the solution of this in water acidified with a little acetic acid gives with silver nitrate a yellow precipitate; with borax and salt of phosphorus a colored bead, in the oxidizing flame colorless to yellowish-green, according to the proportions of vanadium, and a fine green in the reducing flame.

Acidified solutions of vanadic acid in the absence of other coloring materials are yellow to orange.

Hydrogen Sulphide: Turns the solutions blue.

Sulphurous Acid: Same reaction.

Alcohol: Same reaction.

Oxalic Acid: Same reaction.

Zinc: Colors the solutions at first green and later blue.

Ammonium Sulphide: Gives a red color, due to sulpho-vanadate.

Hydrogen Peroxide: Gives a rose or dark red color.

Ether and Turpentine: Give same reaction.

Tannin: Reacting in a neutral solution on a sulpho-vanadate

gives a black precipitate if there is 0.1 percent V_2O_5 present; with 0.01 percent there is produced a black coloration; with 0.001 percent there is produced a distinct bluish coloration. This reagent is very effective as a qualitative test for vanadium. The substance to be tested is fused with sodium carbonate, leached with water, neutralized, and the test made with the tannin solution.

SULPHO-HYPO-VANADATES (*vanadium as acid*).

Red solutions of sulpho-hypo-vanadates are precipitated by dilute solutions of acids, giving a brown precipitate of vanadyl tri-sulphide, $V_2O_2S_3$. The complete precipitation is difficult to attain by simple acidification; it is more completely attained by using a soluble salt of copper.

COLOR REACTIONS OF VANADIC ACID.

Matignon investigated methods for colorimetric determination of vanadic acid, and made the following observations:

(1) Tannin gives a dark blue coloration or precipitate, according to whether the solution is weak or strong in ammonium meta-vanadate, the coloration being very characteristic. A solution in ammonium salt containing 182 milligrams of V_2O_5 per liter takes on a fine blue tint when solid tannin is put into it. One drop of this solution evaporated to dryness on a porcelain capsule and treated with a dilute solution of tannin gives a very distinct coloring. In this way Matignon was able to detect vanadium in a single drop of solution containing only 2 milligrams of V_2O_5 per liter. The edge of the drop on the porcelain becomes marked by a blue line. This is the limit of sensitiveness of the method.

(2) Matignon investigated other substances which had what he called phenolic properties, like tannin. Resorcin, hydroquinone, gaiacol and phloraglucine gave no coloration with vanadic salts; gallic and pyrogallic acids, on the contrary, produced extremely dark blue colorations, the sensibility of which is of the same order as that produced by tannin. These colors become feebly greenish when the liquid is heated to boiling, but regain their intensity and initial tint on cooling.

Pyrocatechin gives also a violet blue coloration many times less sensitive than the preceding.

(3) Pyrogallic acid with an active ether is far more sensitive than the preceding. A solution containing 2 milligrams of vanadium per liter does not give a clear coloration with any of the preceding reagents, but if ordinary ether is added with pyrogallic acid there appears after agitation a reddish-brown coloration. All the commercial ethers do not give the reaction, some producing no coloration, and others with different coloring intensity; there thus appears to be more or less "active" ethers in this respect, and some are inactive. The active ether gives a rose coloration distinctly visible in a solution of ammonium vanadate or vanadic acid containing 0.1 milligram of V_2O_5 per liter; using a few cubic centimeters of this solution in a small test tube, one can easily detect the presence of 1/20000 of a milligram of V_2O_5. Tannin and gallic acid give no reaction at this dilution.

(4) Matignon queried whether the active property of the ether was not vinylic alcohol or ethinol, $CH_2.CH.OH$, the presence of which Polleck and Thummel noted in certain commercial ethers. Experiments verified the accuracy of this premise. Five different ethers, three of which were active as above described, were treated with the Polleck and Thummel reagent (solution of $HgCl_2$ and $KHCO_3$, which gives a white precipitate with ethinol) for half an hour, the two inactive ethers not forming the least trace of precipitate; the active ethers, on the contrary, gave precipitates, the more rapidly the more active they had shown themselves. Moreover, potash is known to destroy ethinol by converting it into a resin. The active ethers immediately turned yellow on addition of potash and lost their activity.

Polleck and Thummel also observed that water removes vinylic alcohol from ether. The active ethers washed with distilled water lost their activity. The facts seem to show that the test for vanadium is dependent on the presence of ethinol. When one of the active ethers is allowed to evaporate in the air the ethinol becomes concentrated in the residue and the activity of the ether is increased; with this ether of increased activity a distinct dark coloration may be obtained in solutions containing only 1/50 milligram of V_2O_5 per liter.

(5) The active ethers may be produced by the aid of ozone. Two inactive ethers, one anhydrous the other ordinary, traversed for an hour by a current of ozonized oxygen do not become immediately active; there was formed, as Berthelot has shown, ether dioxide. However, left to themselves after this treatment for a year they were found to have become active.

(6) Ozonized turpentine, formaldehyde and acetaldehyde do not replace ethinol in this reaction. Ferric chloride and chromates, which also cause this coloration with pyrogallic acid and active ether, must be separated from vanadic acid before applying this test.

QUANTITATIVE ESTIMATION OF VANADIUM.

Two cases are presented in analysis: One may have the vanadium as alkaline vanadate in the presence of excess of alkali, or as sulpho-vanadate with alkaline sulphide or sulph-hydrate.

(1) *Estimation in Alkaline Vanadates.*

Precipitation as ammonium vanadate: Acidify slightly with nitric acid, add ammonia in excess and crystals of ammonium chloride to saturation. Metavanadate of ammonium, NH_4VO_3, almost insoluble in the ammoniacal solution, deposits slowly. In 24 hours the precipitation is complete. Wash by decantation with an ammoniacal saturated solution of ammonium chloride, then by alcohol; filter, dry at 100°, free from the filter, burn it separately and calcine the whole very slowly up to fusion in a small porcelain capsule; weigh as V_2O_5 (56.2 percent V).

This method contains numerous sources of error. The metavanadate is not entirely insoluble and the precipitation is not complete; the solution must be concentrated if only one precipitation is made. During calcination the crucible must be kept uncovered, but as soon as the precipitate melts the calcination must be stopped to avoid loss by volatilization. Further, some potassium may be partially precipitated with vanadium.

Instead of estimating as V_2O_5 one may transform it into V_2O_3, by subjecting the ammonium meta-vanadate with the ashes of the filter to the action of pure dry hydrogen and heating up to bright redness. This operation is not more exact than the preceding except that one can see by a more or less agglomerated appear-

ance whether more or less alkali has been retained in the precipitate.

Precipitation as hypo-vanadate of ammonium: Berzelius gives a method of acidifying the solution, reducing it by oxalic acid and precipitation of the blue solution by ammonia; the ammonium hypo-vanadate is washed in ammoniacal water, and transformed into V_2O_5 by ignition in air.

Precipitation as lead vanadate: In the case of an alkaline vanadate with excess of alkali or alkaline carbonate, acetate of lead may be used as a reagent (Roscoe). In acetate solution there is complete precipitation of lead vanadate; but the precipitate is not of constant composition and therefore cannot be utilized directly. After filtering it out it is washed with water feebly acid with acetic acid, dried at 100° and weighed. The precipitate is then redissolved in nitric acid, using as small a quantity of acid as possible; Add a slight excess of sulphuric acid. Evaporate to white fumes and dilute after cooling. The vanadic acid is thus obtained in solution and the lead precipitates. From the weight of lead sulphate obtained, calculate to PbO, and deduct from the weight of lead vanadate previously obtained, the remainder being V_2O_5.

Corminboeuf proposes a modification of the preceding method by dissolving the precipitate in water acidified with nitric acid, precipitate the lead by a small excess of sulphuric acid, add water, filter and evaporate the filtrate in a small porcelain capsule, finally raise the temperature sufficiently high to drive off all water and acids. At low red the vanadic acid melts, and is weighed directly as V_2O_5.

Precipitation as mercuric hype-vanadate: If the vanadium is present as hypo-vanadate it may be precipitated by mercuric chloride and ammonia. The precipitated hypo-vanadate. mixed with chloro-amidure is calcined in the air. The residue is re-dissolved in ammonium carbonate to separate the last traces of mercury. After filtration and evaporation the residue is finally calcined and weighed as fused V_2O_5.

Precipitation as barium vanadate: This method of Carnot consists in adding ammonia to the acid solution in very slight excess. There is obtained a yellow coloration which disappears rapidly on boiling. To the hot solution barium chloride is added

and the white precipitate left to settle. On testing that no further precipitation takes place on addition of more reagent, the odor of ammonia should be very feeble. The flask being corked it is cooled rapidly. All the vanadic acid is precipitated as barium bi-vanadate, $2BaO.V_2O_5$, containing 37.37 percent V_2O_5 or 21 percent V. The filtrate contains only traces of vanadium. This may be proved by the presence of a brown color on the addition of ammonium sulph-hydrate.

If the boiling after the precipitation of the barium vanadate is carried on too long, the precipitate concentrates and attaches itself to the sides of the flask becoming very difficult to detach and collect.

Strontium salts do not give the same reactions. Treated in the same manner they give no precipitate, either hot or cold. Under the same conditions phosphates or arsenates are precipitated by strontium, which serves as a basis for separating them from vanadic acid. The same reaction will also separate molybdic and tungstic acids from vanadic acid, at least in an approximate manner, for these are precipitated on boiling feebly ammoniacal solutions, but the precipitation is not absolutely complete.

Precipitation as uranium vanadate: Carnot found that uranium salts completely precipitate vanadic acid, not only in ammoniacal solutions but also in the presence of small amounts of free acetic acid, similarly to the property of precipitating arsenic and phosphoric acids. To utilize this reaction the solution is nearly neutralized by ammonia, then several grams of ammonium acetate added and a sufficient quantity of uranium nitrate. The whole is brought to boiling. A yellow precipitate comes down resembling arsenic sulphide. To be sure that the reagent is in excess a drop of the solution is tested by a drop of potassium ferrocyanide, which will give a brown coloration with a uranium solution if it is present in excess. The precipitate is filtered out, washed with pure water, dried and calcined, the filter paper being burned separately. The precipitate dried at 100° is ammonium uranyl-vanadate, $(NH_4)_2U_4V_2O_{12} + H_2O$. Ignited in the air to redness it loses water and ammonia and becomes bright yellow. It is then $U_4V_2O_{11}$, containing 24.22 percent V_2O_5.

This method of determination succeeds in the presence of alkalies and alkaline earths, and also with most metallic oxides

whose acetates do not decompose on boiling. It is especially useful for separating manganese zinc and copper from vanadium, which accompany it in many natural vanadates. The uranium salt gives no separation from phosphoric and arsenic acids. These are entirely precipitated, as are also to a certain extent molybdic and tungstic acids.

2. *Estimation in Sulpho-Vanadates.*

In the absence of any other fixed salt the vanadium may be evaporated to dryness and calcined with ammonium nitrate to transform into vanadic acid. The residue is dissolved in dilute nitric acid and the vanadium determined by the preceding methods.

Sulpho-vanadates and sulpho-hypo-vanadates may be decomposed by hydrochloric acid, strictly avoiding excess. The liquid is allowed to stand under an atmosphere of hydrogen sulphide in a closed flask. Acid is added until the yellow color disappears. It is then decanted, and the liquid tested with ammonium sulphhydrate, which should give no brown color. Precipitation being then complete the precipitate is filtered out, washed immediately with cold water and dried. The precipitate must not be more or less dried and then washed, since the washings will contain vanadium because of the alteration of the precipitate.

After drying, the precipitate is removed from the filter, the latted burned by itself, and the whole ignited in a porcelain crucible, heating gently to burn off sulphur, then moistening with nitric acid, evaporating, drying, and calcining up to fusion; weigh as V_2O_5.

COMPARATIVE DETERMINATIONS.

W. Heike tested several materials by four principal methods. The methods were:

(1) Attack by alkaline carbonates, solution in hot water, separation of iron oxide by filtration, reduction of V_2O_5 to V_2O_2 by boiling with zinc and sulphuric acid, titration by permanganate.

(2) Separation of iron by ether and comparison of the coloration produced by hydrogen peroxide with the coloration given by a known vanadium solution.

(3) Solution of iron by very dilute hydrochloric acid in absence of air, neutralization by barium carbonate, to separate iron and manganese, the residue of chromium and vanadium being fused with alkaline carbonate and nitre. The fusion is taken up with hot water, filtered, and the chromium separated from vanadium by ammonium gas, after having reduced by hydrochloric acid and alcohol and the addition of a few c.c. of ammonium phosphate solution. Chromium is precipitated as phosphate and may be estimated volumetrically. Vanadium passes into the filtrate as sulpho-salt on addition of ammonium sulphide and is precipitated again as sulphide by the addition of nitric acid. The precipitate is calcined to V_2O_5 in the air.

(4) *Method of Campagne:* This method of analysis of metallurgical vanadiferous products was given to the "French Academy of Sciences" at their session October 12, 1903. It is based on the following facts: (1) Ether acidified with hydrochloric acid dissolves ferric chloride, but does not dissolve vanadium oxy-chloride. The vanadium oxy-chloride is reduced to $VOCl_2$ by prolonged boiling with hydrochloric acid while ferric chloride is not altered. Transforming the chlorides into sulphates there is obtained blue di-vanadyl sulphate, $V_2O_2(SO_4)_2$ and ferric sulphate. With permagnate solution one can titrate the di-vanadyl sulphate to vanadic sulphate $V_2O_2(SO_4)_3$ and thus estimate the vanadium. The following are the reactions:

$$V_2O_5 + 6HCl = 2VOCl_2 + 3H_2O + Cl_2.$$
$$2VOCl_2 + 2H_2SO_4 = V_2O_2(SO_4)_2 + 4HCl.$$
$$5V_2O_2(SO_4)_2 + 2KMnO_4 + 8H_2SO_4 = 5V_2O_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O.$$

In determining vanadium in steel 5 grams of the same are attacked with 60 c.c. HNO_3 of Sp. Gr. 1.20, added in small portions. Evaporate on the sand bath, heating strongly at the end so as to transform the nitrates into oxides. Redissolve in 50 c.c. strong hydrochloric acid. The chloride solution obtained is extracted with ether by means of the Carnot apparatus for the method of Rothe. Almost all the iron is retained by the ether. The aqueous solution contains all the vanadium and other metals:

nickel, copper, chromium, etc., and a small quantity of iron. The ether is removed at a low temperature and then the solution is concentrated. To the residue is added 50 c.c. strong hydrochloric acid and it is again evaporated. This operation transposes $VOCl_3$ into $VOCl_2$. To assure the completion of the reduction, boil three times, in the presence of a large excess of hydrochloric acid. After the last evaporation, 5 c.c. of pure concentrated sulphuric acid is added, and the heating continued to white fumes. Let cool, dilute to 250 to 300 c.c. with hot water, and there results a more or less strongly blue solution. Silica is contained in suspension. If this silica is in considerable quantity it may be separated by filtration. The liquid is then titrated by permanganate solution, completing at 60°; the end point is very clear and the rose coloration persists for a long time. The strength of the permanganate solution (containing about 1 gram of crystallized salt per liter) is determined by means of a sodium vanadate solution prepared from a known weight of pure V_2O_5. The reduction may be done either by hydrochloric acid, as described above, or with sulphurous acid.

The same method is applicable to the analysis of ferro-vanadium; if the proportion of vanadium exceeds 25 percent, it is not necessary to remove the iron before titrating. In this case the liquid which has been titrated for vanadium may be reduced again by hydrogen sulphide and re-titrated by permanganate, giving a value corresponding to the V + Fe present, and it is thus easy to determine the amount of iron.

In the case of cupro-vanadium, the alloy is dissolved in nitric acid and the copper is determined electrolytically. The residual solution is evaporated to dryness to obtain the vanadium and iron as oxides, whiich are treated as above, but without separating the iron, which is not present in sufficient quantities to interfere.

If the alloy or mineral examined contains chromium it will be determined as vánadium in the above procedure. Campagne promised to describe a method of successive volumetric determination of vanadium and chromium in the same solution.

The results obtained were as follows:

Method	1	2	3	4
Iron ore	0.07	....	....	0.11
Refining slag	1.40	....	....	1.46
Gray cast-iron, 0.14 cr..	0.15	....	0.21	0.21
Steel, 0.02 cr.	....	0.24	0.27	0.27
Steel	....	0.42	0.54	0.54
Ferro-vanadium	....	....	widely differing results	25.80 25.70

It is seen that the method of Campagne gives higher results than the others and that the reduction of V_2O_5 to V_2O_2 is not always complete. The results of methods 3 and 4 agree very well, the conclusion being that the volumetric methods give too low results. Campagne's method was tried on chemically pure vanadic acid, and it was found that the method does not give too high results, at least in the absence of notable quantities of chromium. In such a case the method 3 may be used, while Campagne's method is applicable in the other cases.

Method of D. T. Williams.

This rests on the reaction described by Sutton, *i. e.*, that V_2O_5 is reduced to V_2O_4 by ferrous solutions: $V_2O_5 + 2FeSO_4 + H_2SO_4 = V_2O_4 + H_2O + Fe_2(SO_4)_3$. One to two grams of ore is treated with 6 c.c. of nitric acid; after complete decomposition evaporate to dryness and heat residue with 4 c.c concentrated sulphuric acid until nitric acid is removed. Dilute with water, bring to boiling and filter out lead sulphate. Oxidize the filtrate with 4 c.c. nitric acid, neutralize the excess of acid with ammonia and re-acidify with sulphuric acid. Cool to 40° and titrate with 0.1 N. solution of ferrous sulphate containing free sulphuric acid. This should be standardized by permanganate or dichromate solutions. Potassium ferri-cyanide may be used as an indicator. The whole solution becomes slightly bluish, but this color does not interfere with the indicator colorations.

Alloys may be dissolved in aqua regia, or in hydrochloric acid and potassium chlorate, evaporating to dryness and treating the residue with 6 to 8 c.c. concentrated sulphuric acid, afterwards treating as above outlined for the minerals. To check the results the solution may be titrated back by a 0.1 N. or 0.2 N. perman-

ganate solution until the rose tint persists 20 seconds. The results thus obtained are generally too high.

Method of Truchot.

This is based on electrolysis. The solution containing 12 to 50 milligrams of V_2O_5 in 200 c.c. is heated to 80-85° and electrolyzed with a current of 2 volts and 0.3 ampere, using a cathode of 80 square centimeters. The operation lasts 8-10 hours. The deposit is washed and lightly calcined; it is yellowish brown and transforms itself into small yellowish red masses of V_2O_5.

The electrolyte should be feebly but sensibly alkaline with ammonia. Excess of alkali increases the hardness of the deposit. Hydrochloric acid must be absent. A temperature of 85-89° is necessary. The calcined deposit is very hygroscopic.

In this method molybdenum deposits with vanadium, but the molybdic acid formed on calcination volatilizes completely at a red heat and is thus separated.

ESTIMATION OF VANADIUM IN ALLOYS.

Method of Nicolardot.

Nicolardot noted that the process which Sefstrom used for finding vanadium in Swedish pig iron might be made quantitative. It is known that steel and iron containing vanadium leave, after being attacked by sulphuric or hydrochloric acids, a black residue of graphitic carbon containing silica and some vanadium. If in this operation all tendency to oxidation is avoided, vanadium remains entirely in the residue in the metallic state; it is not possible to find the least traces either in the supernatent liquor or in the gases produced during the attack. This gives the foundation of a method of determining vanadium in alloys which are attacked easily by hydrochloric acid.

To avoid oxidation it is best to use hydrochloric in preference to sulphuric acid, and to use it in just sufficient quantity to dissolve the alloy. A mixture of $2FeCl_2 + 2HCl$ in the presence of the oxygen of the air may act like ferric chloride. The flask used should be closed with a stopper provided with a valve, and the solution should take place in a current of CO_2. The alloy may also be attacked by the double chloride of potassium and copper, which has been used by Carnot and Goutal for studying the com-

bination of certain elements in cast iron, soft iron and steel. Alloys which are easily attacked by hydrochloric acid are treated as follows:

The finely divided alloy is attacked by hydrochloric acid, density 1.17, diluted with five times its weight of water, or by the double chloride of potassium and copper. To one gram of alloy take 5 c.c. of acid or 5 grams of the salt in solution. On heating two or three hours the attack is complete. Filter and wash well to separate all the iron. Dry, calcine in a platinum crucible, remove silica with a few drops of hydrofluoric acid, heat to 350° to constant weight. The following results have been obtained by the use of this method:

Fusion with soda.	Attack by hydrochloric acid.	Attack by double chloride.
0.13	0.15	0.14
0.20	0.20	0.18
0.41	0.36	0.36

Alloys which are not easily attacked by hydrochloric acid may be dissolved by nitric acid and any copper present separated by electrolysis. The residual solution is evaporated to dryness. redissolved with a few drops of sulphuric acid, and treated by hydrogen sulphide. Continuing to pass hydrogen sulphide, several minutes after the start ammonia is added in just sufficient quantity to precipitate iron, alumina, etc. Filter rapidly with ordinary precautions, evaporate the filtrate to dryness, and keep the sulpho-vanadate at 350° in a weighed crucible until it reaches constant weight. Weigh as V_2O_5.

Method of Matignon and Monnet.

Vanadium is brought to the state of V_2O_4 sulphate, which is oxidized to V_2O_5 sulphate by potassium permanganate. The reduction is done by sulphur dioxide in sulphuric acid solution. The excess of gas is removed by boiling. The solution is diluted strongly and titrated while boiling. The end point is clear and persists. Intermediate tints of a fine blue before complete oxidation give indications of the nearness of the end point.

Method of Jaboulay for Ferro-Vanadium.

Vanadium alloys frequently contain vanadium nitride, which is unattackable by acids; the most practicable means of complete

attack is fusion with sodium peroxide. One advantage of this fusion is that the vanadium is converted into sodium vanadate, which is soluble in water, leaving the iron an insoluble residue. The fusion is usually performed in an iron or nickel crucible. The metal of the crucible is strongly attacked, even at low redness. The fusion is taken up with water and boiled; filtration to remove the insoluble oxides being somewhat slow, there is some advantage in making up the solution to a certain volume and taking a given volume of the clear, filtered liquor for the given test.

Operating as above, low results are obtained, in nickel crucibles 1½ to 2 percent too low, and in iron crucibles as much as ½ percent. This is caused by the oxides of iron and nickel formed retaining a certain quantity of the vanadate of soda. It may be avoided by prolonged boiling of the residue, particularly a second boiling with fresh water.

SEPARATION OF CHROMIUM AND VANADIUM.

Method of Nicolardot.

The separation of chromium and vanadium is very difficult when these two elements are present in large quantities and in about equal portions. The separation is almost impossible by ammonium sulph-hydrate, and very laborious with ammonia. L'Hote's method is not easy of application. The most elegant method is that of Matignon and Bourion, in which the action of sulphur is substituted by that of carbon, but it requires complicated apparatus.

To Nicolardot is due the idea of removing the chromium as chloro-chromic acid. This is attained by treating in a dry flask by fuming sulphuric acid, an anhydrous mixture of the chlorides, chromates and vanadates of the alkalies. The reaction disengages considerable heat; the disengagement of the chromyl chloride is facilitated by using suction, and even by heating towards the end with an alcohol lamp. The last vapors coming off are swept out by a current of air. The attack is so energetic that a little vanadium may be carried over mechanically. This may be intercepted by a small tube containing a few drops of concentrated sulphuric acid. The fuming sulphuric acid used should contain

only a little SO_3 to prevent the chromium chloride being decomposed by water formed in the reaction.

The compound containing chromium and vanadium is fused with a mixture of chlorate and carbonate ($4KClO_3.Na_2CO_3$), the chlorate being in large excess. Small quantities of iron and manganese are separated, if present, by ordinary methods. The alkaline solutions are concentrated, dried and fused. The crucible is washed completely by fusing in it more chlorate, until the chlorate shows no trace of color. If iron is absent, the mixture may be melted immediately in the small flask used for the attack. At the free end of the flask is placed a small washing bulb containing a few drops of concentrated sulphuric acid, followed by a flask containing a dilute solution of soda, and the whole connected with suction. The apparatus is washed out by a current of dry air; then there is run into the reaction flask a few drops of sulphuric acid containing sulphur trioxide. The reaction commences at once, and as soon as it subsides suction is applied, more sulphuric acid added, and dry hydrochloric acid gas is allowed to enter. The flask is finally heated slowly, and also the washing flask, until they no longer give off reddish vapors.

The whole operation is terminated in a few minutes. Care must be taken not to heat too strongly; 60° is sufficient. One tests for complete removal of the chromium by heating on the steam bath and adding sodium sulphide and ammonia in excess to the flask and wash bottle. The precipitate of chromium oxide, if it is abundant, still contains vanadium and must be retreated in the same way again. The vanadium may be entirely separated in the first operation if it is carefully done.

To estimate it, alcohol is added to the liquor, and it is kept on the steam bath until the alcohol has been entirely removed, after which it is titrated by permanganate. The presence of molybdenum does not interfere with this volumetric estimation.

Separation by Means of Ferric Oxide: If the compound containing vanadium and chromium contains large quantities of iron, the separation of the two elements is still more simple.

The compound is attacked by hydrochloric acid, the solution oxidized by nitric acid or chlorate, then evaporated on a steam bath in the presence of an excess of hydrochloric acid. I have proven that under these conditions the larger part of the acid is

removed and there is formed a condensed precipitate of ferric oxide rich in the metalloids. The metals remain in solution.

Sesqui-oxide of chromium brought by any method into the condition of containing less than two molecules of water is changed when heated, at least in part, to the state of chromic acid. The chromium, because of its easy oxidizability, forms chromic acid and passes as made into the condensed precipitate of ferric oxide. The chromium may thus be removed, down to traces, if the temperature is kept not higher than the steam bath, and by moistening the iron deposited before treatment with hot water and several drops of dilute alcohol. The precipitate is detached from the crucible and boiled with water and ammonium sulphate. Under these conditions the chromium is entirely re-dissolved from the precipitate, while the vanadium remains entirely within the precipitate, from which it can be subsequently removed by washing, first with ammonia, followed by fusion with alkaline salts, to remove the least traces. The vanadium is then determined volumetrically.

These two methods made possible the separation of vanadium from other metals and metalloids.

Separation of Arsenic, Vanadium and Molybdenum (Fricdheim, Decker and Diem): Arsenic and molybdenum are precipitated from sulphuric acid solution by hydrogen sulphide; filter and wash on the filter. Evaporate the filtrate on the steam bath and precipitate the vanadium by permanganate, after having removed the excess of hydrogen sulphide. Re-dissolve the precipitate on the filter by a few drops of bromine water, remove excess of bromine by evaporation, finish the oxidation by nitric acid, and precipitate the arsenic by magnesia mixture. A single precipitation will be sufficient if there is not more than 9 molecules of MoO_3 to 1 of As_2O_5.

If more molybdenum is present, too high results will be obtained because of the precipitation of basic sulphate of magnesium, and it is necessary to precipitate the arsenic a second time. The filtrate from the first precipitation of the arsenic may be used for the determination of molybdenum. It is heated slightly with some ammonium poly-sulphide, then acidified with hydrochloric acid, filtered on a Gooch crucible and weighed as MoO_3.

The arsenic may also be separated by distillation, by boiling

a solution of the three salts in the presence of iodide of potassium and hydrochloric acid in a 100 c.c. flask of the Bunsen-Finkener apparatus. For each gram of the salts, 1 to 1.5 grams of potassium iodide and 70 c.c. hydrochloric acid, sp. gr. 1.19, is used. Commence with a low flame in order that the sublimed iodine may not stop up the delivery tube, and gradually increase until the distillation of the arsenic is completed at the end of twenty minutes. Filter out the iodine by glass wool and evaporate with chlorine water to a syrupy consistency, take up with water, evaporate in a platinum crucible, and keep at 40°-50° for half an hour. Let the crucible cool in a desiccator containing P_2O_5 and weigh in a well-closed weighing flask.

Volumetric Determination of Molybdic and Vanadic Acids (Glasmann).

When vanadic and molybdic acids are present in the same hydrochloric or sulphuric acid solution, reduction by zinc gives V_2O_2 and Mo_5O_7, the latter on contact with air passing into Mo_2O_3. Reduction by magnesium gives V_2O_3 and Mo_2O_3. Titration of these bodies may be made with permanganate.

Reduction of 0.25-0.50 grams of the material by zinc on the one hand and by magnesium on the other hand is made by adding 40-45 c.c. hydrochloric acid, sp. gr. 1.19, upon a steam bath, in flasks furnished with Bunsen valves, and requires 1-1½ hours. The titration follows in a 500 c.c. porcelain casseroie after adding 10 grams of manganous sulphate and 300 c.c. boiling water free from air; 0.05 N. permanganate solution should be used. The calculation is made as follows:

If the solution reduced by the zinc consumes x c.c., and that reduced by magnesium y c.c., the difference z corresponds to the oxidation of V_2O_2 to V_2O_3. Three times the difference corresponds to the oxidation of V_2O_2 to V_2O_5, while x minus 3 z corresponds to the oxidation of Mo_2O_3 to MoO_3.

Estimation of Uranium and Vanadium (Finn). The titration of uranium with permanganate after reduction by zinc in sulphuric acid solution gives good results if the reduction is complete, but takes 15-30 minutes and a solution of permanganate not stronger than 0.05 N. However, in a complex mixture of salts, or in a mineral, it is difficult to bring the uranium to

proper purity for titration, and especially so in the presence of vanadium. The uranium, however, may be precipitated in alkaline solution as phosphate, which is not gelatinous and is easy to wash.

A quantity of the ore not containing more than 0.25 grams of U_3O_8 is dissolved in sulphuric acid (1.5) and evaporated to white fumes. Cool and dilute. Boil with an excess of sodium carbonate until the precipitate is well collected. Filter and wash with hot water. Redissolve in the least possible sulphuric acid. Add excess of sodium carbonate, boil, filter, wash. Reunite the filtrates and wash waters, and acidify them with sulphuric acid; add 0.5 grams ammonium phosphate, heat to boiling, and add ammonia to alkaline reaction. Boil a few minutes, filter, wash with hot water containing a little ammonium sulphate.

The filtrate contains the vanadium and is acidified with sulphuric acid; reduce by sulphurous acid to a blue solution, drive off the excess of sulphurous acid by boiling, and titrate with permanganate.

The uranyl phosphate precipitate is redissolved in sulphuric acid and reduced by zinc at least 30 minutes. Separate the zinc by filtration upon asbestos, using suction, and titrate at 60° by permanganate. The vanadium and uranium values of the permanganate may be calculated from its iron value by multiplying by 0.9159 and 2.133 respectively.

Method of D. T. Williams: This rests on the reaction indicated by Sutton, according to which vanadic acid is reduced to vanadium tetra-oxide by ferrous solutions:

$$V_2O_5 + 2FeSO_4 + H_2SO_4 = V_2O_4 + Fe_2(SO_4)_3 + H_2O.$$

Two grams of ore are treated with 6 c.c. of nitric acid. After complete decomposition, evaporate to dryness and treat residue with 4 c.c. concentrated sulphuric acid and evaporate to complete expulsion of nitric vapors. Dilute with hot water and bring to boiling. Lead sulphate precipitates and is filtered off. Oxidize completely with 4 c.c. nitric acid. Neutralize the excess of acid by ammonia, and add sulphuric acid. Cool to 40° and titrate the vanadium with 0.1 N solution of ferrous sulphate containing free sulphuric acid, which has been standardized by 0.1 N dichromate

or permanganate solutions. Potassium ferri-cyanide is used as an indicator. Each gram of iron in the titrating solution corresponds to 0.914 grams of vanadium.

Estimation of tungsten, chromium, nickel, molybdenum and vanadium in steel. (Svennsson).

Dissolve two grams of the steel in 50 c.c. hydrochloric acid, oxidize by boiling with 5-10 c.c. of nitric acid, evaporate almost to dryness, boil with hydrochloric acid (1:5) and filter. Ignite the residue, remove silica by hydrofluoric acid, and weigh the tungstic acid. Concentrate the filtrate which contains the chromium, nickel, molybdenum and vanadium, add a little hydrochloric acid, and remove the larger part of the iron and molybdenum by ether in a Roth apparatus. Evaporate off the excess of ether, boil with 20 c.c. hydrochloric acid, oxidize the potassium chlorate, boil and dilute to 250 c.c., add 10 grams ammonium chloride, and precipitate by ammonia while boiling. Filter, wash with hot water, and titrate nickel in the filtrate, the titration not being interfered with because of the absence of the vanadium.

To eliminate the rest of the vanadium from the precipitate, which contains all the chromium, redissolve in hydrochloric acid and dilute to 150-200 c.c. Add ammonium or sodium phosphate in excess and heat to boiling, and precipitate by ammonia. Filter and wash with hot water. The precipitate. being free from vanadium, is dissolved in 75-100 c.c. sulphuric acid (1:6). Heat to boiling, oxidize by permanganate in slight excess, which is decomposed by boiling, adding if necessary a few crystals of manganese sulphate. Dilute to exactly 500 c.c. per gram of material under treatment. Add excess of standardized sulphuric acid, and titrate the excess with permanganate, which gives the chromium. The precipitate of chromium always contains a little vanadium as manganous vanadate, which is recognized by the quick disappearance of the rose color after titration because of the reoxidation of the permanganate by the vanadic acid formed by the ferric sulphate present. The presence of vanadium in the solution after titration may also be detected by the brown coloration produced by hydrogen peroxide.

Thet nickel is titrated by potassium cyanide. Neutralize the filtrate from the separation of chromium by hydrochloric acid,

add two c.c. concentrated sulphuric acid, or an equivalent quantity of ammonium sulphate, and neutralize by 2 N. ammonia containing 150 c.c. ammonia (density 0.91) per liter, and add 20 c.c. in excess. Add 5 c.c. potassium iodide solution (40 grams per liter) and 5 c.c. silver nitrate solution (1 gram per liter) and titrate with potassium cyanide solution (22.5 grams per liter). The end point is the disappearance of the white cloudiness of silver iodide as seem against a black background. From the quantity of cyanide solution used deduct the quantity corresponding to the silver nitrate solution added, and calculate nickel from the difference. The cyanide solution is standardized by the use of metallic nickel.

To determine vanadium and molybdenum, dissolve 4 grams in hydrochloric acid, oxidize by nitric acid, and filter out tungstic acid. Heat the filtrate and almost neutralize by soda, without forming a precipitate or a permanent red color. Pour the hot solution through a small funnel with constant agitation into an almost boiling solution containing 10 grams caustic soda in 200 c.c. water. After cooling, dilute to 500 c.c. in a graduated flask, and filter on a dry filter paper. Chromium and nickel are precipitated with the iron, while all the vanadium and molybdenum pass into the filtrate. Take exactly 250 c.c. of the filtrate, representing half the material under treatment, and heat almost to boiling, acidify slightly with hydrochloric acid, neutralize with a slight excess of ammonia, add 20 c.c., heat to boiling, and boil until the solution becomes white. Precipitate the vanadium by manganous chloride, avoiding a large excess and keeping the liquid hot, but not boiling. The vanadium may be obtained directly by calcination and weighing as manganese vanadate, but it is difficult to obtain this compound free from manganese oxides in excess, and the following operation is preferable: Allow the precipitate to settle over night in the cold, filter and wash 4 or 5 times with warm water. Any cloudiness in the filtrate is due to manganese oxides containing only traces of vanadium, and may be neglected. Molybdenum is determined in the filtrate. The precipitate is a mixture of manganese vanadate and oxides of manganese. It is ignited with the filter at a low temperature, and then dissolved in a little hot hydrochloric acid. The latter is removed by evaporation with sulphuric acid, diluted, more sul-

phuric acid added, then several c.c. of a solution of sulphurous acid; boil to complete disappearance of the sulphur dioxide odor, titrate hot with permanganate until the rose color persists several seconds. The vanadium is 0.915 times the iron value of the permanganate.

The filtrate separated from the vanadium is rendered acid with excess of hydrochloric acid, boiled until free from chlorine which may form, nearly neutralize with ammonia, and precipitate by lead acetate solution while boiling. Add immediately 50 c.c. concentrated ammonium acetate solution and continue boiling several minutes. Settle, filter, wash with hot water, ignite at a low temperature, and weigh as lead molybdate.

UNIVERSITY OF CALIFORNIA

INDEX

PAGE

Advance Copies of Papers, Regulations Concerning..... 26
Advances, Recent, in Electrochemical Analysis—E. F. Smith..... 65
Advertisements in the Bulletin, Regulations Concerning..... 27
Air-Saltpeter, Manufacture of—Schönherr.....131
Aldrich, R. C.—*Discussions*.....120, 256
Alloys, Copper-Iron, Physical Properties of—C. F. Burgess and J. Aston 241
Analysis, Electrochemical, Recent Advances in—E. F. Smith..... 65
A New Electrolytic Method for Explosive Antimony—R. C. and L. S. Palmer 79
A New Method of Measuring Mean Thermal and Electrical Conductivities of Furnace Electrodes—Carl Hering.....317
Antimony, Explosive—R. C. and L. S. Palmer..... 79
Arc, the Titanium—W. S. Weedon.....217
Arsem, W. C.—*Discussion* 301
Armstrong, Sir H. E—*Discussion*..... 76
Aston, J. and Burgess, C. F.—Physical Properties of Iron-Copper Alloys 241
Badger, W. L., and Frary, F. C.—The Preparation of Calcium.....185
Badische Anilin and Soda Fabrik, Manufacture of Air Saltpeter by the Process of—Schönherr.....131
Baekeland, L. H.—*Discussions*.....94, 118 *et seq.*, 235 *et seq.*
Boron, Pure, Preparation and Properties of—E. Weintraub.....165
Burgess, C. F., and Aston, J.—Physical Properties of Iron-Copper Alloys 241
Burgess, C. F., and Hambuechen, C.—Certain Characteristics of Dry Cells 97
Burgess, C. F.—*Discussions*.....107, 119, 256
By-Laws of the Society 21
Calcium Carbide, Preparation of—M. deK. Thompson.....197
Calcium, Preparation of—F. C. Frary and W. L. Badger.....185
Carbon Electrodes, Conductivity of 345, 351
Carbide of Calcium, Preparation of—M. deK. Thompson.....197
Cell, Dry, Tests—F. H. Loveridge.....109
Cells, Dry, Certain Characteristics of—C. F. Burgess and C. Hambuechen 97
Cementizing, Electro—A. Sang.....257
Certain Characteristics of Dry Cells..C. F. Burgess and C. Hambuechen 97

PAGE
Certificates of Membership 27
Characteristics of Dry Cells—C. F. Burgess and C. Hambuechen..... 97
Committee on Dry Cell Tests 121
Conductance, Practical, of Electrolytes—J. W. Richards and W. S. Landis 85
Conductivity, Mean Electric and Thermal, of Furnace Electrodes—Carl Hering 317
Conductivity of Electrodes, Thermal—C. A. Hansen.....350
Conservation of Natural Resources, Resolution Concerning..... 3
Constitution of the Society..... 14
Contribution to the Electric Smelting of Iron Ore—E. R. Taylor.....229
Copper—Iron Alloys, Physical Properties of—C. F. Burgess and J. Aston 241
Copper Refining, Electrolytic, Power for—W. L. Spalding.....125
Development of Water Power, Motion Concerning..... 4
Directory of Members, Alphabetic 28
Directory of Members, Geographic 53
Dry Cells, Appointment of Committee on Testing of.....121
Dry Cells, Certain Characteristics of—C. F. Burgess and C. Hambuechen 97
Dry Cell Tests—F. H. Loveridge 109
Electrical Conductivity, Mean, of Furnace Electrodes—Carl Hering....317
Electric Furnaces, Electrode Losses in—E. F. Roeber.....363
Electric Furnaces, Laws of Electrode Losses in—Carl Hering.....265
Electric Smelting of Iron Ore—E. R. Taylor.....229
Electro-Cementizing—A. Sang 257
Electrochemical Anaylsis, Recent Advances in—E. F. Smith.....65
Electrode Losses in Electric Furnaces, Laws of—Carl Hering.....265
Electrode Losses in Electric Furnaces—E. F. Roeber.....363
Electrode Losses in Furnaces—C. A. Hansen.....329
Electrodes, Mean Thermal and Electrical Conductivities of—Carl Hering 317
Electrolytic Copper Refining, Power for—W. L. Spalding.....125
Electrolytes, Practical Conductance of—J. W. Richards and W. S. Landis 85
Electrolytic Method for Explosive Antimony—R. C. and L. S. Palmer.. 79
Elliott, A. H.—*Discussions*.....122, 256
Explosive Antimony—R. C. and L. S. Palmer..... 79
Faraday Society Transactions, Regulations Concerning..... 26
Frary, F. C., and Badger, W. L.—The Preparation of Calcium.....185
Furnace Electrode Losses—C. A. Hansen.....329
Furnace Electrodes, Mean Conductivities of—Carl Hering.....317
Furnaces, Electric, Electrode Losses in—E. F. Roeber.....363
Furnaces, Electric, Laws of Electrode Losses in—Carl Hering.....265
Geographic Directory of Members..... 53
Gin, G.—Memoir on Vanadium.....393

PAGE
Graphite Electrodes, Conductivity of....345, 351
Grower, G. C.—*Discussions*....301
Guests in Attendance, Sixteenth General Meeting, List of.... 5
Hambuechen, C., and Burgess, C. F.—Certain Characteristics of Dry Cells 97
Hansen, C. A.—*Discussions*....204, 214, 236 *et seq.*, 300, 362
Hansen, C. A.—Furnace Electrode Losses....329
Hering, Carl—*Discussions*....93 *et seq.*, 106, 121, 184, 235, 255, 299 *et seq.*, 325, 352 *et seq.*, 376
Hering, Carl—A New Method of Measuring Mean Thermal and Electric Conductivities of Electrodes....317
Hering, Carl—Laws of Electrode Losses in Electric Furnaces....265
Heumann, E. M., Tucker, S. A., and Kudlich, H. F.—Preparation of Silundum 207
Iron-Copper Alloys, Physical Properties of—C. F. Burgess and J. Aston 241
Iron Ore, Electric Smelting of—E. R. Taylor....229
Kennelly, A. E.—*Discussion*....297
Kohn, W. M.—*Discussion*....214
Kudlich, H. F., Tucker, S. A., and Heumann, E. M.—Preparation of Silundum 207
Landis, W. S., and Richards, J. W.—The Practical Conductance of Electrolytes 85
Landis, W. S., and Richards, J. W.—Translation of Memoir on Vanadium 393
Laws of Electrode Losses in Electric Furnaces—Carl Hering....265
Lidbury, F. A.—*Discussions*....215, 236 *et seq.*, 303, 325
Local Sections, Regulations Concerning.... 24
Loeb, Morris—*Discussion* 93
Losses from Electrodes in Electric Furnaces—Carl Hering....265
Losses From Electrodes in Electric Furnaces—E. F. Roeber....363
Losses From Furnace Electrodes—C. A. Hansen....329
Loveridge, F. H.—*Discussions*....118 *et seq.*
Loveridge, F. H.—Dry Cell Tests....109
Manufacture of Air-Saltpeter—Schönherr....131
Mean Conductivities of Furnace Electrodes—Carl Hering....317
Members, Alphabetical Directory of.... 28
Members Elected and Qualified in 1909.... 8
Members, Geographic Directory of.... 53
Membership Certificates 27
Members in Attendance Sixteenth General Meeting, List of.... 5
Motion Concerning Water Power Development.... 4
Natural Resources, Resolution Concerning.... 3
Ore, Iron, Electric Smelting of—E. R. Taylor....229
Palmer, R. C. and L. S.—Paper on Explosive Antimony.... 79
Papers, Advance Copies of, Regulations Concerning.... 26

PAGE
Parsons, C. L.—*Discussion*.....107
Physical Properties of Iron-Copper Alloys—C. F. Burgess and J. Aston 241
Power for Electrolytic Copper Refining—W. L. Spalding.....125
Power, Water, Motion Concerning Development of..... 4
Practical Conductance of Electrolytes—J. W. Richards and W. S. Landis 85
Preparation and Properties of Calcium Carbide—M. deKay Thompson.197
Preparation and Properties of Pure Boron—E. Weintraub.....165
Preparation of Calcium—F. C. Frary and W. L. Badger.....185
Preparation of Silundum—S. A. Tucker, H. F. Kudlich and E. M. Heumann 207
Proceedings of the Sixteenth General Meeting..... 1
Recent Advances in Electrochemical Analysis—E. F. Smith..... 65
Refining, Copper, Power for Electrolytic—W. L. Spalding.....125
Regulations Concerning Local Sections..... 24
Resolutions Adopted at Sixteenth General Meeting..... 3
Resources, Resolution Concerning Conservation of..... 3
Richards, H. C.—*Discussion*.....304
Richards, J. W., and Landis, W. S.—The Practical Conductance of Electrolytes 85
Richards, J. W., and Landis, W. S.—Translation of Memoir on Vanadium 393
Richards, J. W.—*Discussions*.....94 *et seq.*, 213, 226, 235, 255, 299 *et seq.*, 325, 360.
Roeber, E. F.—*Discussions*.....352, 384
Roeber, E. F.—Electrode Losses in Electric Furnaces.....363
Salom, P. G.—*Discussion*.....234
Saltpeter, Air, Manufacture of—Schönherr.....131
Sang, A.—Electro-Cementizing 257
Schönherr, Dr.—Manufacture of Air-Saltpeter.....131
Sections, Local, Regulations Concerning..... 24
Seede, J. A.—*Discussion*.....107
Sharp, C. H.—*Discussions*.....119 *et seq.*
Sharples, S. P.—*Discussion*.....108
Silundum, Preparation of—S. A. Tucker, H. F. Kudlich and E. M. Heumann 207
Smelting, Electric, of Iron Ore—E. R. Taylor.....229
Smith, E. F.—Recent Advances in Electrochemical Analysis..... 65
Sperry, E. A.—*Discussion*..... 93
Taylor, E. R.—*Discussions*.....234 *et seq.*
Taylor, E. R.—Electric Smelting of Iron Ore.....229
Tests of Dry Cells—F. H. Loveridge.....109
The Manufacture of Air-Saltpeter—Schönherr.....131
The Practical Conductance of Electrolytes—J. W. Richards and W. S. Landis 85

PAGE
The Preparation of Calcium—F. C. Frary and W. L. Badger.....185
The Preparation of Silundum—S. A. Tucker, H. F. Kudlich and E. M. Heumann 207
Thermal Conductivity, Mean, of Furnace Electrodes—Carl Hering....317
Thermal Conductivity of Electrodes—C. A. Hansen.....350
The Titanium Arc—W. S. Weedon.....217
Thompson, M. deK.—Preparation of Calcium Carbide.....197
Titanium Arc, The—W. S. Weedon.....217
Tone, F. J.—*Discussion*.....214
Townsend, Wolcott—*Discussion*..... 92
Transactions, Price of Complete Sets..... 25
Tucker, S. A.—*Discussions*.....213 *et seq.*, 225
Tucker, S. A., Kudlich, H. F., and Heumann, E. M.—Preparation of Silundum 207
Vanadium, G. Gin.....393
Water Power Development, Motion Concerning..... 4
Weedon, W. S.—*Discussions*.....226 *et seq.*
Weedon, W. S.—The Titanium Arc.....217
Weintraub, E.—*Discussion*.....184
Weintraub, E.—Preparation and Properties of Pure Boron.....165
Whitney, W. R.—*Discussions*.....91, 122, 213, 224 *et seq.*
Wire, Electro-Cementizing of—A. Sang.....257
Zinc-Coating Wire by Electro-Cementizing—Sang, A.....257

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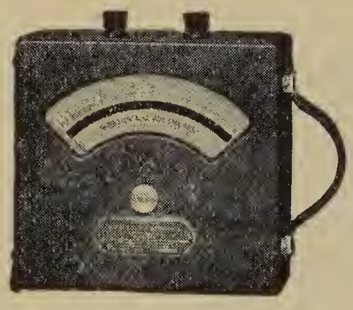

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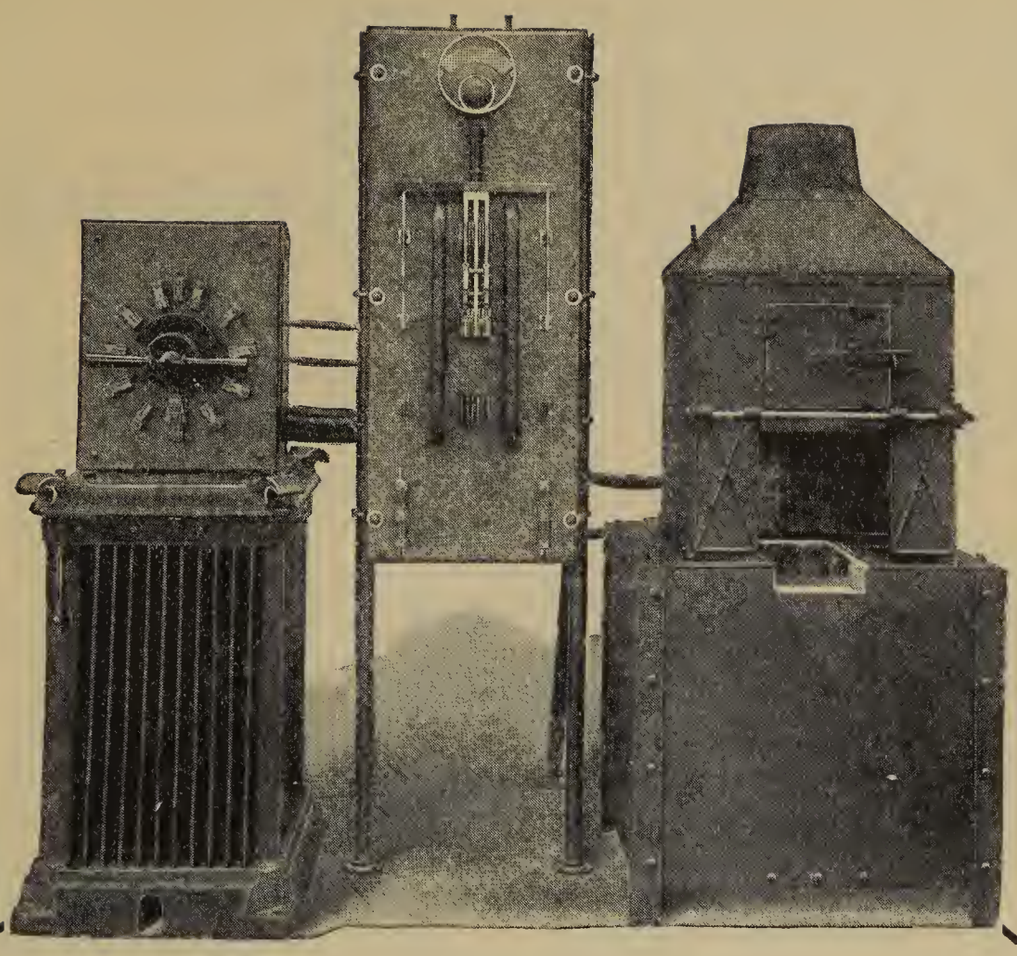

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